PSYCHORHEOLOGY OF SKIN CREAM

Ruth Elizabeth Greenaway

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ABSTRACT

The relationship between physical and sensory properties of 40 model skin creams was investigated. Creams were formulated according to an experimental design to ensure that a wide range of textural properties could be produced from a minimal number of ingredients.

The core project study comprised of objective sensory profiling of model skin creams (QDA, Quantitative Descriptive Analysis) and the physical characterisation of the textural and flow properties relevant to the use of skin creams (rheology, texture analysis and force plate analysis). Sensory attributes related to initial skin cream application procedures (firmness, thickness, resistance, spreadability, stickiness and slipperiness) were highly correlated to rheological and texture analysis parameters. Attributes related to application procedures involving a time factor and absorption of cream into the skin (drying, dragging, final greasiness and absorption) were found to be correlated to parameters from force plate analysis.

A consumer study was also conducted based on a subset of the model skin creams to identify properties of skin creams that are liked by the naïve consumer. Cluster analysis and external preference mapping identified groups of consumers with different types of liking behaviour. The firmness and thickness of the samples were found to be important regarding consumer liking.

Models were generated to predict the sensory properties of creams from the physical parameters. Rheological parameters (G' at 100 % strain and $\log G''$ at 100 % strain) produced the most robust models that could predict firmness, thickness, resistance, spreadability and slipperiness. Models predicting attributes involving absorption of cream into the skin were less robust, these involved force plate analysis parameters.

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ABBREVIATIONS AND SYMBOLS

ABBREVIATION	UNITS	DEFINITION
ANOVA		Analysis of Variance
C		Cream
CA		Cetyl Alcohol
CV	%	Coefficient of Variation
EPM		External Preference Mapping
ex		supplied by/purchased from
HWB		Hot Water Bath
LVD		Linear Viscoelastic Domain
OAS		Oscillation Amplitude Sweep
o/w		oil-in-water
p		statistical significance level
PCA		Principal Component Analysis
PC		Principal Component
QDA		Quantitative Descriptive Analysis
r		Pearson's correlation coefficient
R^2		regression coefficient
SA		Stearic Acid
SD		Standard Deviation
SS		Steady Shear
TA		Texture Analysis
TEA		Triethanolamine
UoN		University of Nottingham
w/o		water-in-oil
w/w	%	weight by weight
YS	Pa	Yield Stress

SYMBOL	UNITS	DEFINITION
A	m^2	Area
a-value	s	Cross time constant
F	N	Force
Fz	N	Load
G'	Pa	storage modulus
G''	Pa	loss modulus
G^*	Pa	complex modulus
h	m	shear gap
p-value	-	Cross rate constant
s	m	deflection path
γ	%	strain
$\dot{\gamma}$	s^{-1}	shear rate
$\tan\delta$	1	loss factor
η	Pa.s	shear viscosity
η_∞	Pa.s	infinite shear viscosity
η_0	Pa.s	zero shear viscosity
η^*	Pa.s	complex viscosity
μ	-	friction coefficient
ν	$m.s^{-1}$	velocity
τ	Pa	shear stress
ω	$rad.s^{-1}$	angular frequency

STRUCTURE OF THESIS

This Thesis is organised as follows:

- **Chapter 1**: General introduction including aims and objectives of this PhD, background information on skin creams and a literature review exploring different techniques that have been used to measure both sensory and physical properties of skin creams.
- **Chapter 2**: Materials and methods employed to investigate the textural properties of the model skin creams including the experimental design, skin cream formulation, physical measurements (rheology, texture analysis and force plate analysis) and sensory techniques (QDA and consumer study).
- **Chapter 3**: Results and discussion in which sensory and physical results are discussed separately.
- **Chapter 4**: The relationship between sensory and physical data is described in this chapter including the development of predictive models from which sensory properties of creams can be predicted using physical parameters. The relevance of these models is discussed in terms of consumer liking.
- **Chapter 5**: In this chapter the work carried out in this PhD has been summarised and suggestions for future work are presented.

1. INTRODUCTION

1.1 CONTEXT

Consumer testing is a key part of the new product development process (Kemp et al., 2009); if a product is to succeed it is important that the consumer likes the product so they will purchase it again (Van Kleef et al., 2005). Consumer liking studies are useful for determining which products consumers like and why they like certain products more than others. However, this is a difficult area of research as consumers do not necessarily know what they want or why (Van Kleef et al., 2005), but they are good at judging whether they like or dislike a product. Therefore other areas of sensory science are used in combination with consumer studies to aid understanding of consumer liking. In order to understand consumer liking, the way a product is perceived needs to be measured. In sensory science as well as affective (consumer) testing, descriptive tests may also be carried out, whereby a group of people (a panel) are trained to rate products for specific attributes; thus they act as instruments allowing quantitative data to be collected (Meilgaard et al., 1999).

Sensory data obtained through trained panel rating can be used to compare with hedonic data to determine which attributes are important to the consumer. Using trained panellists to measure products for sensory attributes rather than instruments provides valuable data as humans are usually more sensitive than instruments (Lawless and Heymann, 1998). Although sensory testing enables a wealth of useful information about a product to be collected, it has the disadvantage of being time consuming and expensive (Stone and Sidel, 2004). Therefore if an instrumental measurement can be identified that provides data that can be correlated to sensory trained panel data then this would be very beneficial (or attractive from an industrial perspective) as it would allow a manufacturer to make predictions about a product’s sensory properties from the instrumental data only, thus saving the manufacturer time and money.

1.1.1 Psychorheology

Psychorheology is the study of the relationship between rheological and sensory properties of a material (Wegener, 1997). This research applies psychorheology to the study of skin creams. Rheological properties play a huge role in the sensory perception of skin cream. Cosmetic products are generally evaluated by touch, whilst a consumer spreads a sample of cream on the skin; subconsciously the brain can appreciate the uniformity of the sample, coherence, absorption capacity, smoothness and its texture in general (Marriott, 1961). Likewise the force required to remove a sample from its container or squeeze the sample out of a tube is judged according to consumer expectation. For example consumers expect a body lotion to be thinner than a cream (Brummer, 2006).

Understanding the relationship between objective rheological measurements and both objective and subjective sensory tests enables a wide range of information about the product to be obtained, including the rheological properties of material that give rise to certain sensory characteristics and sensory attributes that appeal to the consumer. These aspects are all important when developing new products to match consumer expectations (Wegener, 1997). Correlating rheological parameters to sensory attribute data obtained from a trained panel can be achieved through predictive modelling.

1.1.2 Experimental design

In order to produce predictive models a systematic experimental approach must be followed. Response surface methodology allows relationships between measured sensory responses and design factors to be quantified (Design-Expert, 2000). Average results from trained panel rating of samples for different attributes are submitted to a stepwise regression analysis. This yields a predictive equation relating the value of the sensory responses to the independent variables (design factors) (Meilgaard et al., 1999).

Different types of response surface design may be chosen to suit the experiment including factorial and central composite designs. Many of these designs indicate that a large number of experiments must be carried out in order to produce accurate models. D-optimal response surface designs on the other hand calculate the minimum number of experiments (i.e. skin cream compositions) required to produce accurate predictive models (for sensory behaviour) (de Aguiar et al., 1995). This type of design is therefore an appropriate choice in research involving sensory tests where sensory fatigue means that a limited number of samples can be measured during one session and panellist attendance of more than two to three sessions a week is not practical.

1.2 SKIN CREAMS

1.2.1 The skin and its function

The skin is the largest organ of the human body (area ~ 2 m^2). Its main functions include protection of the body, thermoregulation and sensory perception (Winkelmann, 1961; Brummer, 2006; Couturaud, 2009). The skins natural protective system consists of secretions from the sebaceous and sweat glands including moisturising factors, amino acids and lactic acids that cover the surface maintaining a pH between 5 and 6. If acid dominates, then the skin will be dry and feel tight, on the other hand an excess of bases results in oily skin. Therefore an important role of cosmetic emulsions in skin care is to restore the natural balance of acids and bases (Brummer, 2006) thus to obtain (and retain) healthy skin (Shai et al., 2001). Prevention of dry skin can be achieved through application of moisturising creams and lotions which contain combinations of humectants, occlusives and emollients to help maintain hydrated skin. Humectants attract and hold water within the skin thus maintaining hydration and minimising water loss. Examples of humectants include glycerine, urea and lactic acid. Occlusives, such as mineral oil or petrolatum, form a layer on the surface of the skin thereby moisturising the skin through prevention of evaporation of water. Emollients, including lanolin and sunflower seed oil glycerides, provide partial occlusion through hydrating the stratum corneum improving its overall appearance and condition (Shai et al., 2001; Rawlings et al., 2004).

1.2.2 Influence of environmental factors on skin condition

Skin condition is affected by stress, temperature, relative humidity, the menstrual cycle and physical work history (Gee et al., 2005). Therefore, consumer skin cream requirements are likely to change throughout the year depending on the weather and work in which they are involved. Skin creams are often marketed for different skin types including dry, normal, sensitive or oily skin. However, these terms are ambiguous; for example, there is no definition of normal skin, it is diagnosed in comparison with other skin types i.e. a normal skin is not oily, not dry and not mixed (Couturaud, 2009). Skin types can vary in different areas of the body; in particular skin on the hands is more susceptible to damage through repeated hand washing, manual labour or cold weather (Shai et al., 2001). It has been reported that biophysical properties of the skin vary with age, gender, ethnicity and anatomical site but this is an area of continual research in order to understand the complex relationships and reasons for differences (Szabo et al., 1969; Olsen et al., 1995; Manuskiatti et al., 1998; Mussi et al., 1998; Rawlings, 2006; Couturaud, 2009).

The fact that environmental factors can affect skin condition means that great care should be taken when designing experiments involving testing of samples on the skin. Both the preparation of the skin prior to the measurement (i.e. hand washing, the type of soap used, skin temperature) and the instrumental conditions employed throughout the measurement, should be closely controlled and monitored to avoid external factors affecting the test results (Prall, 1973).

1.2.3 Types of skin cream

There are many different types of skin cream: vanishing creams, night creams, cleansing creams, moisturising creams, foundation creams, cold creams and eye creams (Shai et al., 2001). The majority of creams can be described as semi-solid materials and are oil-in-water (o/w) emulsions (aqueous creams). Water-in-oil (w/o) emulsions (oily creams) also exist although these are typically less popular due to their characteristic greasy, oily feel on application to the skin. However, more recently development of emulsifiers has enabled w/o creams of lighter texture to be produced (Epstein, 2009). The first known w/o emulsion was prepared in 150 AD by Galen, a Greek physician, who melted purified beeswax with three or four parts olive oil scented with rose petals. While the mixture was cooling he added as much water as possible to form a smooth cream. This formulation is known as a 'cold cream'[1] and serves as a model for those prepared today (Forster and Herrington, 1997).

1.2.4 Skin cream composition

Skin creams can be formulated from many different ingredients, the most common include: water, emollients, emulsifiers and preservatives. Water is the most commonly used liquid in cosmetic preparations. The level of water in a skin cream can affect the skin feel on application, as water evaporates from the formulation it has a cooling effect on the skin surface. Vanishing creams contain high levels of water which results in easy application of the cream that appears to vanish during application leaving a non-greasy residue on the skin (Shai et al., 2001).

Emollients include oils and lipids that are easy to spread on the skin. They hydrate the skin through partial occlusion of the stratum corneum which improves the overall appearance (Rawlings et al., 2004). Different emollients provide different

[1] Cold creams are pseudo-emulsions i.e. simple mixtures of oil and water with no emulsifier present therefore they are not stable. On application to the skin, the water evaporates quickly creating a cooling effect hence the name cold cream.

skin feels and are therefore selected depending on the function. For example if the cream was to be a night cream, then emollients with a higher degree of oiliness would be appropriate since the presence of a greasy residue on the skin over night is not a problem. Alternatively, hand creams for use during the day would be better suited to lighter oils (Salka, 1997; Shai et al., 2001) that result in a less greasy residue on the skin, which is more appropriate for carrying out daily tasks.

One dilemma faced by the cosmetics industry is the fact that moisturising agents (oils, humectants and thickening polymers) tend to increase the stickiness of products which is undesirable from a consumer's perspective (Kusakari et al., 2003; Kudoh et al., 2006). Therefore careful selection of ingredients is important to ensure that creams with acceptable skin feel are produced. Likewise quality control measures, including sensory and instrumental tests designed to measure the skin feel of products, are key to ensure that products of acceptable skin feel are produced.

Emulsifiers and preservatives are key ingredients regarding the structural and microbiological stability of skin creams. Emulsifiers present in skin cream formulations are often formed from combinations of surfactants and fatty alcohols (Eccleston, 1986). Examples of such ingredients include cetyl alcohol, stearic acid, glyceryl monostearate (fatty alcohols), triethanolamine stearate, sodium stearate and cetrimide (surfactants). These ingredients are often capable of performing more than one role within the formulation e.g. they may be classed as an emulsifiers, stabilisers, thickening agents or emollients (Eccleston, 1997).

Preservatives are necessary in skin creams to prevent unwanted bacteria or fungi spoiling the formulation and potentially putting the consumer at risk (Shai et al., 2001). Also from a consumer's perspective it is convenient to purchase a skin cream that can be used during the course of a year (or longer) without requiring specific storage conditions (e.g. refrigerated storage). Thus it is important to get the combination of stabilisers and preservatives correct to ensure that the overall properties (sensory and physical) of the cream are retained during storage (Al-Bawab and Friberg, 2006). Levels of preservatives considered acceptable for use in skin creams vary depending on the product although the levels are generally low (≤ 1 %). Antimicrobial agents commonly used as preservative systems in skin creams include Phenylmercuric nitrate (0.01 %), Parabenz (0.3 %), and Cetyltrimethylamonium bromide (1 %) (Bloomfield, 1996).

It is very important that ingredient combinations and levels used in cream formulations are thoroughly tested so as not to endanger the consumer. Currently in the EU, regulations are very strict; only cosmetics that comply with detailed regulations set out in Dir. 76/768/EEC and its Annexes may be placed on the European market (Pauwels and Rogiers, 2009) while in the USA the Federal Food, Drug and Cosmetics Act of 1938 still stands (Simion, 2009). Koller (1902) highlights one reason why it is crucial that good checks on the quality of cosmetics are carried out *"vanity, and the desire to conceal personal imperfections, are so dominant in some individuals, as to render them careless whether the articles they use are dangerous to health"*.

1.2.5 Function of skin creams

Skin cream function varies with product type. In general products either act directly on the skin (e.g. moisturisers) or they can act as a delivery vehicle for transferring a specific active ingredient to the skin (e.g. in sun screens, anti acne medicaments) (Epstein, 2009). Skin creams can be categorised into 3 functional groups: Cosmetics improve the appearance and feeling of the skin without impacting significantly on its structure or function; Drugs prevent or treat diseases which can alter the structure and/or function of the skin; Cosmeceuticals are substances with the function of cosmetics (to improve appearance) yet they contain active ingredients to enhance the function of the skin e.g. anti aging products (Griffiths, 2010).

Cosmetic skin creams along with their functions to improve the overall skin feel, must also be appealing to the consumer in their appearance and feel during application. Barry and Grace (1972) reported that the main consumer judgement of cosmetics (excluding therapeutic and cosmetic effects) depends on the 'texture profile' which includes the following characteristics; appearance, odour, extrudability where applicable, initial sensations upon contact with the skin, spreading properties, tackiness and residual greasiness after application. This range of factors that affect consumer judgement highlights the complexity of human perception.

Humans have five main senses, classified by Aristotle as: sight, smell, touch, taste and hearing (Geldard, 1953). Each of these play a role in 'our perception and understanding of the world around us' (Barker, 2009). This PhD focuses on the textural properties of skin creams, which are likely to be perceived mainly through touch perception (although sight, smell and hearing may also play a role). There is doubt over whether touch can be defined as a sense on its own, since the touch sense is affected by other psychophysical factors including pressure, pain, temperature and irritation (Geldard, 1953). These factors along with the influence of environmental factors on skin condition (see Chapter 1.3.2) emphasise the importance of controlled experimental conditions when carrying out sensory tests.

1.3 SENSORY EVALUATION

"Sensory evaluation is a scientific discipline used to measure, analyse and interpret characteristics of materials as they are perceived by the senses of sight, smell, taste, touch and hearing" (Stone and Sidel, 1986). The ability to quantitatively measure the sensory properties of a material is beneficial as it allows comparison between different samples to be made. As mentioned in section 1.3.5, human perception is complex involving a combination of senses in the judgement of products. Therefore sensory science is a useful tool for measuring sensory properties of materials that are difficult to measure instrumentally (Lawless and Heymann, 1998), see also Chapter 1.1.

Sensory evaluation does not guarantee products will be successful as other factors including price, image and packaging play a role in consumer perception of products. However, it is still important to assess sensory properties of products to ensure quality is maintained and that they do not fail due to sensory deficiencies (Stone and Sidel, 1986).

1.3.1 Sensory evaluation methods

Sensory tests may be subjective or objective. The type of test selected depends on the information required from the study. Subjective sensory tests measure consumer preferences (likes and dislikes of the sample) while objective sensory tests are more analytical.

1.3.1.1 Objective sensory methods

Objective sensory methods may be classified as descriptive or discrimination tests. Descriptive sensory test methods include QDA (quantitative descriptive analysis) (Stone et al., 1974), spectrum descriptive analysis (SDA) (Meilgaard et al., 1999) and flash profiling (Dairou and Sieffermann, 2002) whereby panellists involved have been screened for sensory acuity and are trained to rate samples in a specified manner for intensity (objective) rather than liking (subjective). Descriptive tests enable a wide range of information about product characteristics to be gained. This is useful when investigating the relationship between sample sensory properties and other factors such as consumer liking, ingredient concentrations or physical properties. In QDA, the attributes and protocols used for rating the samples are determined by the panellists (Kemp et al., 2009). SDA on the other hand uses attributes selected from a predefined lexicon (Meilgaard et al., 1999). For this reason the attributes used in QDA are thought to be closer to attributes a consumer may use (Kemp et al., 2009). QDA is therefore a more appropriate choice when the aim is to correlate sensory trained panel data with consumer liking data.

Discrimination test methods include triangle tests and paired comparison tests; for these tests assessors may be trained or untrained (Lawless and Heymann, 1998; Kemp et al., 2009). Discrimination tests involve panellists identifying differences between samples. For example in a triangle test the panellist is presented with three samples (two are the same and one is different), and asked to identify the odd sample. This type of test can therefore be used to determine whether two samples are perceptually different or whether two samples are sufficiently similar to be used interchangeably (Meilgaard et al., 1999). In product development or quality control this can be useful as a manufacturer may want to determine whether a difference in formulation can be identified by the consumer.

1.3.1.2 Subjective sensory methods

Various studies can be carried out with consumers to obtain subjective data including qualitative (focus groups, interviews, home use tests, questionnaires) and quantitative methods (hedonic rating, preference tests). Qualitative methods gather information about products from a consumer's perspective i.e. they determine which products are used by consumers and why certain aspects of these products are important to them, while quantitative methods attempt to quantify the level of liking for different products (Lawless and Heymann, 1998). Quantitative methods are therefore more appropriate where the intention is to correlate liking data to sensory attribute properties.

1.3.2 Sensory evaluation of skin creams

Van Reeth (2006) reported that sensory expectations for skin care products are related to culture, age, skin type, gender, setting and climate. Therefore when carrying out consumer studies it is important that information about the consumer is also recorded in case any important links between consumer background (e.g. age or culture) and liking can be identified. This information could be obtained in the form of a questionnaire. Sensory evaluation of skin creams is complicated by the fact that cream is absorbed into the skin therefore any application leaves residue on the skin. This means strict conditions must be in place when carrying out sensory tests involving skin creams to ensure that carry over effects do not occur affecting results.

A few examples of work carried out on cosmetics as reported in the literature are given below. Aust and co-workers (1987) developed an objective method for sensory evaluation of dry skin care products. Results showed that the panel could reproducibly measure relative intensities of product attributes on a numerical scale. Although the skin cream products tested were labelled as having the same dry skin efficacy, it was discovered that there were significant differences in skin cream perception amongst the products. This is of interest, as products labelled to be clinically effective in alleviating dry skin are often not equally well received in the market. Aust et al. (1987) suggested that attributes other than ‘moisturising ability’ might be more meaningful to the consumer. Oiliness, greasiness and residue, for example, may be more important consumer perceptions. Trained panel descriptive profiling data is therefore a good way of bridging the gap between clinical and consumer data.

SDA is another objective method that can be applied to skin creams. Lee et al. (2005) used SDA to evaluate 12 aqua cream products for 26 attributes. The aim was to investigate sensory characteristics of skin care products (lotions and creams) followed by identification of attributes important in aqua cream products using PCA biplot analysis. Some of the attributes were also screened based on consumer opinions for improvements in the aqua cream products on the market. Results suggested that desirable characteristics in these creams are a high degree of wetness, spreadability and moisturisation, and a low degree of stickiness, gloss and oiliness.

Companies carry out consumer studies to determine how the consumer perceives their products. Although valuable data can be obtained from these tests, they are subjective, time consuming and costly (Brummer and Godersky, 1999; Kusakari et al., 2003). Therefore understanding the relationship between consumer liking and product attribute properties is a key step in developing relevant predictive models from which sensory properties of creams can be predicted (see Chapter 1.1.2). Once predictive models are created this would mean that future predictions about skin creams could be made without carrying out the expensive, time consuming consumer studies and trained panel rating.

1.3.3 Relating consumer liking data to product attribute properties

Various statistical analysis methods may be used to aid understanding of the relationship between consumer liking data and product attribute properties (including trained panel rating scores and instrumental data). The most commonly used techniques used are described below.

1.3.3.1 Principal component analysis (PCA)

Principal component analysis (PCA) is a multidimensional statistical technique used to visualise the correlations between variables (XLSTAT, 1995-2009). PCA analyses the correlation between a group multivariate observations (e.g. trained panel attribute ratings for different samples) and identifies the axis along which maximum variability occurs (Meilgaard et al., 1999). This is referred to as the first principal component (PC1). The second principal component (PC2) is the axis along which the greatest amount of remaining variability lies. PC2 is orthogonal to PC1. Further principal components may also be included, however typically 2 – 3 principal components are sufficient to explain 75 – 90 % of the total sensory variability in a data set (Meilgaard et al., 1999).

PCA results are plotted in the form of a multidimensional map (Kemp et al., 2009) showing the correlation between variables (attributes or parameters analysed) known as a correlation circle. The relative locations of the samples measured can be superimposed onto a PCA map (called a PCA biplot) thus allowing the difference between samples to be clearly seen as well as the sample groupings (Wegener, 1997). PCA is therefore a useful tool for visualising the correlations between attributes rated by a trained panel that would be difficult to interpret from raw data alone. It is also useful for identifying which attribute properties prevail in certain samples more than others, thus highlighting the reason for differences between samples.

1.3.3.2 Cluster analysis

Cluster analysis is multivariate statistical method that identifies groups of observations based on the degree of similarity or dissimilarity in their ratings (Meilgaard et al., 1999). This technique can be used to analyse data from consumer studies to identify different groups of liking behaviour. Consumer liking responses for various products are transformed into a similarity or dissimilarity matrix and then a clustering algorithm is applied to the matrix to identify clusters of consumers with similar liking behaviour (MacFie, 2007). These algorithms may be hierarchical or non-hierarchical. Once an observation has been assigned to a cluster by hierarchical methods, it cannot be moved to another cluster. However, non-hierarchical methods allow movement of observations between clusters (Meilgaard et al., 1999).

Hierarchical methods may be agglomerative or divisive. Agglomerative methods consider each observation (in this case each consumer) to be in a cluster of their own and the analysis successively merges the observations until one cluster exists. Divisive methods occur the other way round whereby one cluster containing all consumers is broken down into separate groups until each individual consumer is a separate cluster (Meilgaard et al., 1999). Results are presented in the form of a dendrogram which allows the analyser to select how many clusters to analyse further (MacFie, 2007). Different algorithms may be chosen to determine the distance of (dis)similarity between clusters. The most commonly used hierarchical algorithms are average linkage (also termed the unweighted pair group average linkage) and Ward's method (1963). The former joins two clusters at the average (dis)similarity level between all possible combinations of cross-cluster pairings. The latter finds the maximum ratio of between cluster to within cluster variance (MacFie, 2007). This method tends to produce clusters of similar size, which is beneficial in further analysis as it allows conclusions drawn about individual clusters to be more relevant.

In non-hierarchical methods, the observations are moved from one group to another iteratively, starting from an initial partition. The number of groups is specified prior to analysis (Fraley and Raftery, 1998). The most common non-hierarchical method is the k-means method whereby each observation is assigned to a cluster based on its distance from the centre of a cluster, as more observations are added the distance from the centre changes hence the cluster groupings change. This process is therefore repeated until no further changes occur (Meilgaard et al., 1999).

1.3.3.3 Preference mapping

Preference mapping is a widely used technique that involves applying cluster analysis (see Chapter 1.4.3.2) to consumer liking data to determine different groups of consumers, then relating this to trained panel data through internal or external preference mapping (MacFie, 2007). Internal preference mapping is a variant of PCA, it uses consumer preference ratings (internal data) to locate the samples on the map (Meilgaard et al., 1999); perceptual data (trained panel data) may then be fitted into the map allowing the relationship between consumer liking and attribute properties to be visualised. The mathematical procedure of singular value decomposition is used to decompose the matrix of consumer preferences into its basic structure which consists of two matrices: 1) a preferential product map where the products are positioned in the lower dimensional products space (products x dimensions) and 2) individual consumers' preference weights along the dimensions of the product map (consumers x dimensions) (Van Kleef et al., 2006). Trained panel attribute ratings can then be fitted to the product map (products x attributes), through calculating the correlation coefficient of the panel sensory means with the PC scores, then using these correlations as coordinates to plot the variables on the graph with the range -1 to +1 on each axis (MacFie, 2007).

Internal preference mapping assumes consumers focus on a single or highly correlated group of sensory attributes and rate their liking in relation to how much or how little of these attributes are present. This type of liking behaviour follows a vector model trend. Vector model consumers show a liking trend in one direction therefore by moving in the direction of the vector, the consumer will like the sample more. The length of the vector is proportional to the goodness of fit (MacFie, 2007).

On the other hand, external preference mapping uses external data (trained panel data) to build a product map based on attribute ratings; consumer preferences are then fitted to the map at a later stage (Van Kleef et al., 2006). In this case PCA is applied to trained panel data (attribute rating scores) to decompose the matrix of perceptions into its basic structure of the two matrices: 1) a perceptual product map where the products are positioned in the lower dimensional products space (products x dimensions) and 2) sensory attribute weights relative to the dimensions of the product map (attributes x dimensions) (Van Kleef et al., 2006). Consumer liking scores are then plotted in relation to the product space, this allows the pattern of consumer liking behaviour to be visualised, see Figure 1.1. Note that the plot is two dimensional to aid illustration.

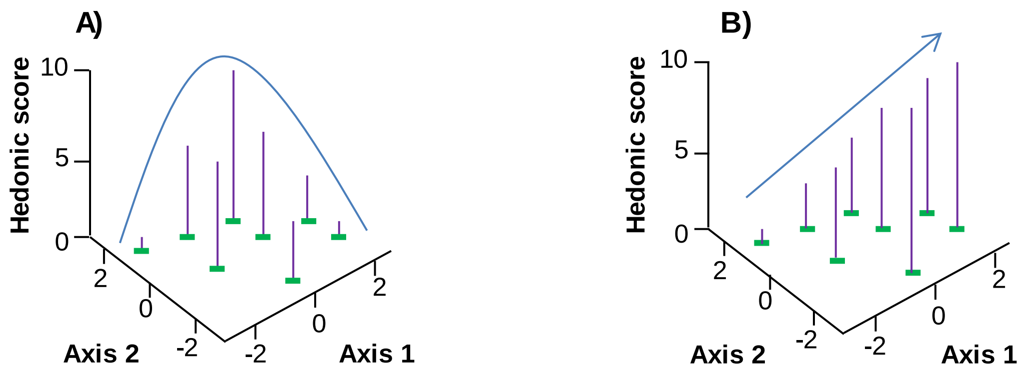

Figure 1.1: *Examples of typical patterns of consumer liking behaviour where = the products, = consumer liking data and represents the pattern of liking behaviour. A) Indicates the pattern of liking behaviour for a consumer with an ideal point model while B) represents a consumer with a vector model*

External preference mapping may be referred to as PC response surface methodology (RSM) since effectively RSM analysis of overall liking, cluster overall liking or each respondents liking scores separately is carried out (MacFie, 2007). The different patterns (or surfaces) of consumer liking behaviour may be described in terms of models, these include vector models, ideal point, anti-ideal point and saddle point models. Vector models were described previously regarding internal preference mapping. Ideal point models represent consumers that like samples in a specific area 'ideal point' of the sensory space; moving outside that area, the consumer will no longer like the sample. Anti-ideal point models represent consumers with a clear idea of what they do not like (opposite of ideal point) and saddle models include consumers who show an ideal point in one direction but a vector model in the other (MacFie, 2007). There are various types of ideal model including circular (the liking space is in a circular relationship around the ideal point), elliptical (the space around the ideal point liked by the consumer is encompassed by an elliptical shape) and quadratic (a more complex relationship which includes interaction terms). Note that the elliptical and quadratic forms of the model also apply to anti-ideal and saddle point models and the circular form also applies to the anti-ideal point model (XLSTAT, 1995-2009).

As well as preference maps showing the location of consumers in relation to the different samples and attribute properties, in external preference mapping, contour plots are also generated. These highlight the areas in the design space that are liked by different proportions of consumers, thus indicating the popularity of different samples and the significance of different attributes in liking. At each point on the chart, the percentage of judges for whom preference calculated from the model is greater than their mean preference, is calculated. This allows the regions of high and low preference within the design space to be identified and highlighted using a colour scheme: hot colours (e.g. red) represent a high proportion of consumers with high preferences and cold colours (e.g. blue) represent a low proportion of consumes with high preferences (XLSTAT, 1995-2009).

Overall, internal preference mapping tends to provide a greater understanding of consumer preferences since the product space is defined by consumer responses. External preference mapping on the other hand captures more of a product understanding as the product space is defined by attribute ratings (Van Kleef et al., 2006; MacFie, 2007). Therefore the method chosen for analysis will depend on the aims and objectives of the research.

1.3.3.4 Kano modelling

An alternative method for looking at consumer liking data was proposed by Kano (1984). This method, known as Kano's model of customer satisfaction, looks at satisfaction and dissatisfaction scores separately. The idea being that the relationship between satisfaction and dissatisfaction is not linear; rather the overall satisfaction depends on how well a consumer's needs are fulfilled (Riviere et al., 2006). The model proposes three types of product attributes that could affect consumer satisfaction: "must be" attributes; performance attributes and attractive attributes. Figure 1.2 illustrates the relationships for these attributes:

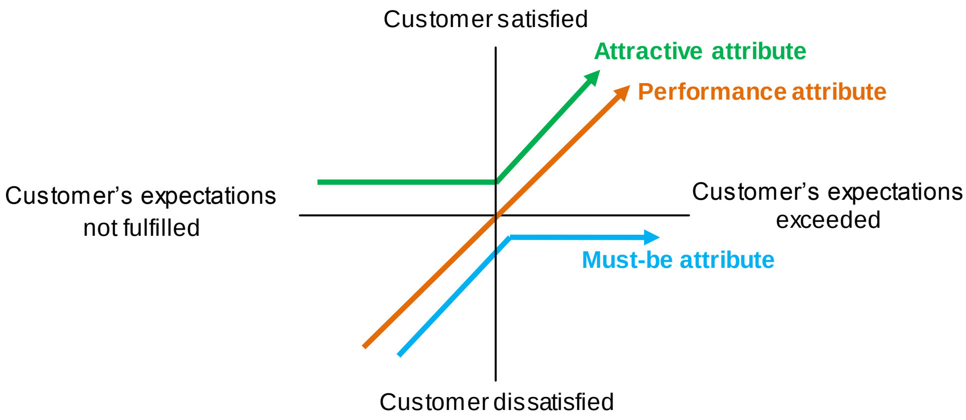

Figure 1.2: *Kano's model of customer satisfaction (Matzler and Hinterhuber, 1998)*

"Must be" attributes are very important, they relate to the basic requirements of the product. If these attribute requirements are not fulfilled, the consumer will be very dissatisfied (Riviere et al., 2006). However at the same time, fulfilling these requirements does not increase liking, it just fulfils the customers' expectations. For example for certain skin cream products (Nivea) it is expected that there will be a foil fresh seal under the lid. If it is not there, the consumer will be dissatisfied; if it is there, it will go unnoticed.

Performance attributes may satisfy or dissatisfy the consumer depending on their level of fulfilment. The higher the level of fulfilment, the higher the customer satisfaction and vice versa (Matzler and Hinterhuber, 1998). For example when buying a car, the greater the mileage per gallon, the more satisfied the consumer.

Attractive attributes have the greatest influence on consumer satisfaction (Riviere et al., 2006). If attractors are present they can lead to greater consumer satisfaction, however if these attributes are not present the consumer will not be dissatisfied. For example vocal recognition with a mobile phone can lead to high customer satisfaction but if it is lacking the customer will not be dissatisfied.

Products containing attractive attributes that exceed customer's expectations can lead to a high level of customer satisfaction which in turn can lead to customer loyalty that can enhance a company's market share (Matzler and Hinterhuber, 1998). However, as technology advances, attractive attributes may become performers and eventually as people take them for granted they may even become must be attributes.

1.4 RHEOLOGY

1.4.1 Introduction and definitions

The term rheology comes from the Greek words *rheo* (to flow) and logos (science) (Fischer, 1948) hence rheology is literally ‘flow science’ and is often defined as the study of flow and deformation of materials (Dickinson, 1992; Macosoko, 1994; Brummer, 2006; Mezger, 2006). Studying the rheological properties of skin creams allows a range of information about the product to be gained. For example when a consumer removes a sample of cream from its container it will generally hold its structure as it is transferred to the skin. As the consumer spreads the cream on their skin, the sample will be deformed. The forces and speeds involved in spreading will affect the cream behaviour. Rheology allows these forces and speeds to be measured enabling an understanding of cream behaviour under different conditions to be gained. Rheological properties relevant in the characterisation of the flow properties of semi-solids will be defined in the following text and relevance of these parameters to skin creams will be discussed in Chapter 1.5.2.

Fundamental rheological parameters can be defined in terms of a parallel plate model (see Figure 1.3) in which the lower plate is stationary and the upper plate with area, A, is set in motion by a shear force, F. The resulting velocity, ν, is measured. The distance between the plates, h, is referred to as the shear gap and the sample is sheared within the gap. The distance the upper plate moves, the deflection path, is equal to s. This model assumes that the sample adheres to both plates and does not slide or slip and that flow conditions are laminar (flow in the form of layers) rather than turbulent (Mezger, 2006). Note that the conditions between plates in a rheometer (instrument designed to measure rheological data) are not as simple as in the parallel plate model. However, if the shear gap is narrow enough, the necessary requirements are met and the model can be used to describe standard rheological terms.

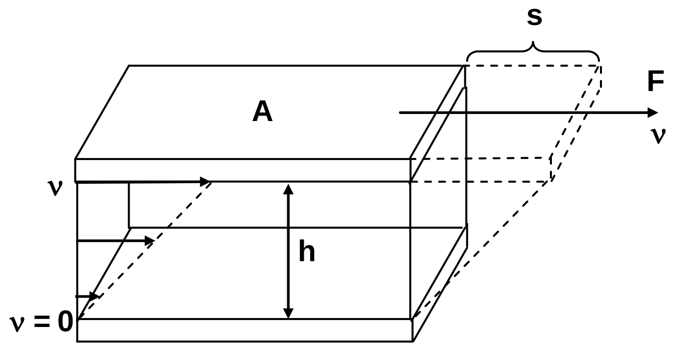

Figure 1.3: *Parallel plate model used to define fundamental rheological parameters (Mezger, 2006)*

The **shear stress, τ (N.m^{-2} = Pa),** is the force per unit area when the force acts parallel to the surfaces and is given by:

$$\tau = \frac{F}{A} \qquad (1.1).$$

The **shear rate, $\dot{\gamma}$ (s^{-1}),** is the ratio of the velocity of the plates to the shear gap:

$$\dot{\gamma} = \frac{\nu}{h} \qquad (1.2).$$

The **viscosity, η (Pa.s),** is a measure of the resistance of a material to flow and is calculated by:

$$\eta = \frac{\tau}{\dot{\gamma}} \qquad (1.3).$$

The **strain, γ (dimensionless units),** is a measure of the shear deformation and is defined by:

$$\gamma = \frac{s}{h} \qquad (1.4).$$

The **storage modulus, G′ (Pa),** is a measure of the deformation energy stored by a sample during an oscillatory shear process. Materials that store the entire deformation energy exhibit complete reversible deformation behaviour therefore on removal of the force, the sample returns to its original structure (elastic behaviour). The G′ therefore provides an indication of the elastic behaviour of a material.

The **loss modulus, G″ (Pa),** on the other hand is a measure of the deformation energy dissipated by the sample during an oscillatory shear process. Materials that lose energy show irreversible deformation behaviour, therefore, on removal of the force these samples remain deformed (viscous behaviour). The G″ provides an indication of the viscous behaviour of the material.

The **loss factor, tanδ (dimensionless),** gives the ratio between viscous and elastic portions of the material, thus providing an indication of the viscoelastic behaviour of the material:

$$\tan\delta = \frac{G''}{G'} \quad (1.5).$$

1.4.2 Rheology of skin creams

Various rheological tests can be applied to characterise cosmetic creams including shear stress or shear rate sweeps, creep recovery tests, and oscillatory stress and frequency sweeps (Brummer and Hamer, 1997; Korhonen et al., 2001; Korhonen et al., 2002; Ribeiro et al., 2004; Brummer, 2006). In industry however evaluating the quality of cosmetic products to ensure consistency between different batches is more commonly achieved through use of simple measurements such as comparing the apparent viscosities measured using a Brookfield viscometer (Marriott, 1961; Hopkinson and Williams, 2007). In quality control simpler measurements are preferred as they are quicker to measure and therefore more efficient for comparing the quality of cream as it is produced. In contrast measurements made using a rheometer usually involve a longer test duration followed by data analysis to extract the relevant parameters and are therefore more time consuming and less suitable for use in quality control.

1.4.2.1 Viscosity and shear rate

Understanding the flow behaviour of skin creams is very important from an industrial perspective. Power consumption, blending time and ability to pump the product through pipes in a factory depends on the viscosity of the product which can change during processing depending on the temperatures and shear applied to the product (Wibowo and Ng, 2001). During scale up from laboratory to factory production, for example, if the sample is too thick, then problems may occur when pumping it through the factory or filing it into containers. Ward et al. (1974) reported a case when equipment broke down during the filling stage of shampoo manufacture due to the shear thickening properties of the shampoo that were initially overlooked.

Flow properties of creams also influence how easy it is to apply the sample to the skin and how well it will spread on the skin. Ideal rheological characteristics of skin creams have been described as including a low viscosity at high shear so it is easy to apply (i.e. it flows readily at high shear so it can be rubbed into the skin) and a relatively high viscosity at low shear rates so that it does not spill easily (Forster and Herrington, 1997; Wibowo and Ng, 2001). Viscosity is often used to monitor formulation stability over time (Van Reeth, 2006). If it changes dramatically over time this suggests the sample is unstable.

Shear rates relevant to the application of skin cream are argued to range between 10 – 100,000 s^{-1} (Henderson et al., 1961; Langenbucher and Lange, 1969; Barry and Grace, 1972; Wibowo and Ng, 2001) while typical shear rates in processing of skin creams include: 500 – 1000 s^{-1} pouring from a bottle and 100 – 10,000 s^{-1} extrusion from a bottle or tube (Barry and Grace, 1972; Wibowo and Ng, 2001).

1.4.2.2 Yield stress

The yield stress is a measure of the stress required to induce flow in a product, which is an important property of skin creams. For skin creams the magnitude of the yield stress relates to the strength of the interparticle interaction in the three-dimensional network microstructure (Adeyeye et al., 2002). Consumers tend to associate a 'cream' with a reasonably structured material i.e. a substance with a noticeable yield stress, whereas a lotion is generally expected to be less viscous showing limited yield behaviour (Brummer, 2006). The yield stress along with the viscosity provide an indication of how easy it will be to distribute a sample on the skin (Colo et al., 2004) and are therefore likely to influence consumers when purchasing a cream. For example, if a consumer wanted a hand cream to protect their hands during work involving contact with water and/or cold conditions, then a cream with a high viscosity and yield stress would be beneficial. These properties ensure that the cream remains thick on application providing a protective barrier layer to coat the hands (Colo et al., 2004). Alternatively, if the cream was for use on the body, a less viscous sample may be more beneficial as it is likely to cover a larger area more quickly (Brummer and Godersky, 1999).

Barnes and Walters (1985) argue that a yield stress does not actually exist; they suggest that the yield stress only defines what cannot be measured i.e. if a material flows at a high stress, it will also flow at low stresses (even if this flow is very small). Although this theory is plausible, practically it is useful to be able to measure the properties of material therefore in this research the yield stress will be measured.

1.4.2.3 G′, G″ and tanδ

G′ and G″ provide an indication of the flow behaviour of a material. If G′ is greater than G″ this shows that elastic behaviour is dominating (tanδ < 1) and samples are in the gel (or solid) state. If on the other hand G″ is greater than G′ (tanδ > 1), this shows that viscous behaviour is dominating and samples are in the liquid state. Measuring tanδ values at different strains (under different levels of shear deformation, γ) therefore provides an indication of the spreading properties of the sample (the point at which the sample will spread easily). This is thought to be related to the absorption properties of a sample given that a sample must spread well on the skin prior to absorption (Adeyeye et al., 2002). Values for tanδ also provide an indication of the internal structural strength of the cream, the smaller the tanδ values, the stronger the internal structure (Colo et al., 2004).

1.5 TEXTURE ANALYSIS

1.5.1 Introduction

Texture may be defined as a multiparameter attribute that is affected by structure (molecular, microscopic or macroscopic) and sense of feel or pressure. Certain aspects of texture may also be detected by the eyes (e.g. thickness of a cream in a container) and the ears (e.g. the sound produced when biting a cracker) (Szczesniak, 1990). Textural properties of materials are important as they can affect processing, handling, influence habits, affect shelf-life and consumer acceptance of products (Stable-Micro-Systems, 2005).

The texture of materials may be characterised by sensory or instrumental methods. In some cases, instrumental methods are favoured as they are relatively rapid and cheap compared to sensory evaluation and they can be carried out under more strictly controlled conditions. If an instrument could be designed to predict consumer acceptance or to replace a sensory descriptive panel, this would have huge beneficial implications for industry (save time and money). Therefore a large amount of research over the past 100 years has gone into the development of instrumental methods for evaluating texture or consistency (Szczesniak, 1987).

Many instruments have been designed to specifically measure the textural properties of materials including the Lee-Kramer shear press (meat, peas, apple, cheese), General Foods Corporation Texturometer (wide range of food products, cake biscuits, toffee, turnip) and the Instron tensile tester (potato, potato chips, apples) (Sherman, 1970). In this research the TA.XT plus Texture Analyser (Stable-

Micro-Systems) was used. A range of different probes and fixtures may be attached to this texture analyser for measuring a vast array of different materials including cosmetics, food products, adhesives and packaging. Types of texture analysis measurement include: compression, puncture and penetration, cutting and shearing, extrusion, tension, fracture and bending/snapping and adhesion (Stable-Micro-Systems, 2005). Texture analysis tests described above are commonly used in industry for quality control measures as they are quick, relatively cheap and easy to use (Szczesniak, 1987; Paoletti et al., 1995).

1.5.2 Texture analysis of skin creams

In this PhD, the back extrusion test was used to characterise the textural properties of skin creams (see Chapters 2.4.2 and 3.4). This test measures the consistency, firmness, index of viscosity and cohesiveness as a probe is inserted into and withdrawn from a sample (Stable-Micro-Systems, 2005). These parameters are relevant to the properties of skin cream as they provide an indication of the structural properties of the material which can influence the performance of the product. For example, how well it can be applied to the skin and how well it will absorb. This is particularly important in the pharmaceutical industry as the spreading properties influence the ease of absorption which in turn affects efficiency of drug release (Woolfson, 1997).

The consistency can provide an indication of how the cream will behave in different types of packaging i.e. whether it will easily squeeze out of a tube and whether on squeezing the product will continue flowing or whether it will break sharply (Stable-Micro-Systems, 2005). The firmness may be crudely related to the sensory firmness a consumer perceives on dipping their finger in a cream prior to application i.e. it is a measure of the deformation experienced by the sample. The index of viscosity and cohesiveness are measured as the probe is withdrawn from the sample. They can provide a measure of the extent of structural recovery following application of shearing stress (the effect a consumer's finger scooping cream out of a container has on the remaining cream) (Woolfson, 1997). The cohesiveness and index of viscosity values will also influence the sample spreading properties thus indicating how easy it will be to spread the sample on the skin which is likely to affect the rate of absorption (Woolfson, 1997; Adeyeye et al., 2002).

1.6 FRICTION AND TRIBOLOGY

1.6.1 Introduction and definitions

In the 15th century the main facts about friction were recorded by Leonardo da Vinci (1452 – 1519) who wrote: "Friction produces double the amount of effort if the weight is doubled" and "The friction made by the same weight will be of equal resistance at the beginning of movement although the contact may be of different breadths or lengths" (Naylor, 1955; Comaish and Bottoms, 1971).

The laws of static friction known today were written by Amontons in 1699 while Coulomb distinguished between static and kinetic friction in 1781, showing that the latter was usually less than the former (Comaish and Bottoms, 1971). Amontons' laws state that the friction force is 1) independent of the nominal area of contact between the surfaces and 2) directly proportional to the load, therefore if the load is doubled the friction force is also doubled. This second law can be expressed mathematically as follows:

$$F = \mu L \tag{1.6}$$

where F is the friction force, L is the load and μ the friction coefficient (μ = F/L) (Gohar and Rahnejat, 2008). Amontons' laws are true for dry contacting surfaces whereas in the presence of lubricants (for example on applying cream to the skin) the friction force is not always directly proportional to the load and the friction coefficient can change with velocity (Sivamani and Maibach, 2006). For example, if there is a thick film of fluid between the surfaces, the frictional properties will depend entirely upon the physical properties of the interposed layer between the surfaces (Naylor, 1955).

Tribology is the scientific study of interactions between contacting surfaces in relative motion. It is a multidisciplinary subject incorporating 3 disciplines: Friction (physics and mechanical engineering), Lubrication (mechanical engineering and chemistry) and Wear (materials science) (Gohar and Rahnejat, 2008). Different regimes of lubrication can be identified through analysis of Stribeck curves in which the friction coefficient is plotted against the sliding speed (speed/load) or film thickness (film thickness/roughness height) (Dresselhuis et al., 2007). An example of a Stribeck curve is given in Figure 1.4.

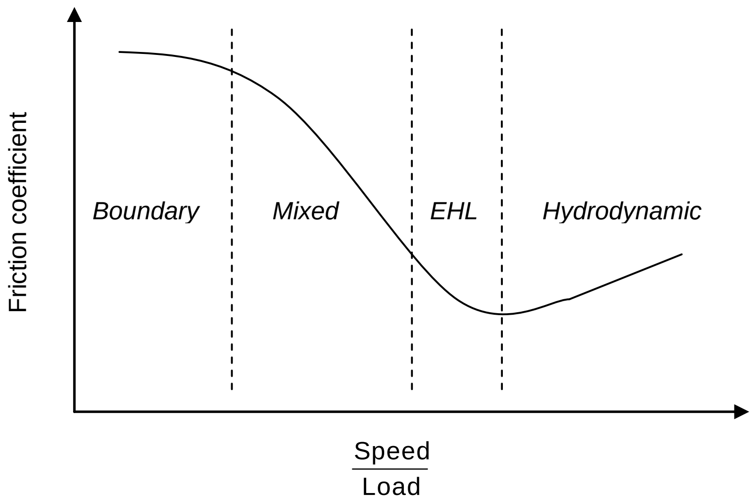

Figure 1.4: *Stribeck curve illustrating the regimes of lubrication, where EHL refers to the elastohydrodynamic lubrication regime.*

The lubrication properties of different materials are of interest for a variety of reasons. For example biological polymer solutions are used as lubricants in synovial joints and contact lenses (Vicente et al., 2005). Understanding the extent to which these solutions form boundary or hydrodynamic films has huge implications in producing products of maximum comfort to the consumer. In many technological applications the aim is to optimise lubrication of the surfaces in order to minimise wear and reduce energy consumption. For this reason the **elastohydrodynamic**

lubrication (EHL) and **hydrodynamic lubrication regimes** are often of most interest (Nishikawa and Kaneta, 2006; Dresselhuis et al., 2007). In these regimes, the high speeds cause a build up of hydrodynamic pressure which separates the surfaces. This means that the load on the interface is entirely supported by the fluid film/lubricant between the surfaces thus resulting in low friction and no wear. Therefore the ability to form a hydrodynamic or EHL film depends mainly on the viscosity of the lubricant (Dresselhuis et al., 2007). In the hydrodynamic regime the film thickness between contacting surfaces may be thicker than that in the EHL regime due to higher speeds causing greater hydrodynamic pressure and therefore further separation of the surfaces. The friction coefficient can also increase in the hydrodynamic regime (see Figure 1.4) due to higher speeds creating fluid drag on the moving surfaces (Anderson et al., 2008).

On the other hand during skin cream application, the thickness of the cream sample layer changes from thick to thin (Brummer, 2006). Therefore, although initial application procedures may relate to EHL or hydrodynamic lubrication, it is likely that later stages of application would be related to the mixed or boundary lubrication regimes. **Boundary lubrication** occurs when low speeds and high loads are acting between surfaces; together these factors lead to a high contact area between surfaces with a small level of fluid being present at the interface, thus resulting in high friction (Gohar and Rahnejat, 2008). As the speed increases or the load decreases, the surfaces will begin to separate allowing a thin fluid film to form between them (Anderson et al., 2008). This leads to a drop in friction that can be defined as the **mixed** lubrication regime (in this regime there may still be some surface-surface contact but the higher level of fluid present between the surfaces causes a drop in overall friction coefficient) (Anderson et al., 2008; Gohar and Rahnejat, 2008).

During the boundary and mixed lubrication regimes, the friction properties depend on the characteristics of the interacting surfaces (Dresselhuis et al., 2007) whereas in EHL and hydrodynamic lubrication the friction properties depend entirely on the lubricant properties (Naylor, 1955). Understanding the frictional properties of skin and the effect application of skin cream has on skin friction may therefore be important in understanding consumer perception of skin feel.

1.6.2 Frictional properties of skin

Naylor (1955) was the first to study friction in relation to skin in any detail. In his paper in 1955 he made the following statement: “Friction is one of the commonest insults to which human skin is exposed”. Skin friction depends on age, anatomical site, skin hydration and skin health (Sivamani and Maibach, 2006). Tribology is a convenient method for carrying out non-invasive techniques to quantitatively assess skin health and hydration (Sivamani and Maibach, 2006). A large number of skin friction measurements cited in the literature involve the use of a probe either rotating or sliding in linear motion across the skin (Sivamani and Maibach, 2006). Probe materials range from metal e.g. stainless steel or copper (Sivamani et al., 2003) to glass (Koudine et al., 2000; Adams et al., 2007), to nylon (Highley et al., 1977) and polyethylene (Naylor, 1955; Comaish and Bottoms, 1971). Fewer studies investigate the effects lubrication has on the frictional properties of the skin (Loden et al., 1992; Koudine et al., 2000; Kusakari et al., 2003).

In experiments involving a probe moving across the skin the usual site of measurement is the volar forearm as there is little variation in the volar forearm skin across gender, age and ethnicity (Sivamani et al., 2003). More recently studies measuring frictional properties occurring at different speeds and loads between a panellist’s finger and the test material have been reported. Gee at al. (2005) used this technique to measure the frictional properties experienced when stroking natural rubber, three thermoplastic elastomers, polyethylene, a steel block, polycarbonate and glass. Results showed higher friction coefficients than equivalent measurements using a steel probe. Derler et al. (2007) measured the friction occurring between a human finger and various textiles. They found that a polyurethane coated polyamide fleece with a similar surface structure to skin related best to skin under dry conditions.

Hopkinson et al. (2008) have developed these methods further focussing on the applications relevant to personal care products. They measured the frictional properties occurring between a panellist's finger and synthetic skin between which products of differing rheological characteristics (including creams, oils, polymer solutions and Vaseline petroleum jelly) were placed. This method is termed force plate analysis.

1.7 LITERATURE RELATING INSTRUMENTAL AND SENSORY DATA

Investigating the sensory and instrumental properties of skin creams individually provides useful information about the products. However, understanding the relationship between instrumental and sensory properties is also important. If it was possible to identify and measure physical parameters that determine the skin feeling of cosmetic products then this coupled with consumer liking data would place the manufacturer in an advantageous position (see Chapter 1.1). Studies reported in the literature that have attempted to correlate sensory and physical data will now be discussed.

Brummer and Godersky (1999) investigated whether there was any correlation between perception of skin feeling and rheological properties of cosmetic products. The shear stress at the onset of flow, τ_F, was measured via a shear stress ramp in which the shear stress at which the viscosity value was maximum, was taken as the τ_F. It was thought that these values could be correlated to primary skin feeling (sensations at the start of cream application). The results showed poor correlation of τ_F with primary skin feeling; this was explained by the fact that on application of cosmetics to the skin, the product flows rapidly making it difficult for panellists to judge the onset of flow in sensory tests. Stationary viscosity values, η, at typical shear rates involved in the end of skin cream application (up to 10^5 s^{-1}) were correlated to secondary skin feeling (sensations at the end of cream application). The maximum shear rate was estimated using Equation (1.7) where it was assumed that the spreading rate, ν , was 1 $m.s^{-1}$ and the film thickness of the cream, χ , was 0.01 mm:

$$\dot{\gamma} \equiv \frac{\mathrm{d}\nu}{\mathrm{d}\chi} \approx \frac{\Delta\nu}{\Delta\chi} = \frac{1\mathrm{m.s}^{-1}}{10^{-5}\mathrm{m}} = 10^5\,\mathrm{s}^{-1} \qquad (1.7).$$

Viscosities of Newtonian oils were measured and rated by a panel for skin feeling. The viscosities of the cosmetic emulsions were then measured and the results were compared to the oil viscosity of optimal skin feeling. Sensory results showed that the optimal stationary viscosity was 0.024 Pa.s. They found that for lotions the shear rate at which the viscosity was 0.028 ± 0.005 Pa was ~ 5000 s^{-1} and for creams the shear rate was ~ 500 s^{-1}. This was reported to be due to the fact that the shear rate of product application depends on the type of product as well as the fact that skin feeling is product specific. This study also found that the absorption capacity perceptible on the skin increased with decreasing viscosity of the cream.

Wang et al. (1999) measured transepidermal water loss (TEWL) and skin capacitance (SC) values as a function of time after application for 12 skin cream formulations. These creams contained the same basic ingredients but the type and levels of polymer used were varied. A further set of tests involved panellists spreading as much cream as they felt necessary to moisturise both hands followed by TEWL and SC measurements. The panel also rated the samples for 14 attributes. The TEWL and SC values allowed the levels of moisturisation different cream formulations provided to the skin to be compared. Lower TEWL values and higher values for SC signify an increase in water content of the stratum corneum and therefore an increase in moisturisation. Rheological characterisation of these creams suggested that the overall rheology of the skin creams had little effect on the moisturising efficacy or perceived perceptual attributes. It was also observed that the amount of cream panellists used to gain sufficient moisturisation was fairly constant despite the differing rheological properties of the samples used. The type and level of polymer appeared to affect the moisturising efficiency and some of the sensory attributes of the cream but the reasons for these findings were unclear.

Barry and Grace (1972) investigated the rheological conditions that exist during spreading of lipophilic preparations on the skin. They adapted a method developed by Wood (1968) for evaluating the in-mouth shear conditions of liquid foodstuffs. The spreadability of topical preparations ranging in consistency from stiff semi-solids to mobile liquids was judged by a sensory panel via ordinal scaling, ratio scaling, preference testing and by comparison with Newtonian silicone oils. Samples were also characterised rheologically to obtain discontinuous (up curve only) and continuous (up and down curves) shear flow curves. Results were plotted in the form of master curves as log shear stress versus log shear rate. Flow curves for the silicone oils with the closest spreadability properties to each topical preparation (as defined by panellists) were also plotted on the master curve. The intersection of the flow curve of the topical preparations with the Newtonian reference fluid properties provided an estimation of the spreading conditions. The range of shear rates during spreading was found to vary between 400 and 2500 s^{-1}. Consumers preferred the spreading characteristics of samples with viscosities ranging between 0.39 – 1.18 Pa.s, which could be defined by the region 400 – 700 s^{-1} and 200 – 700 Pa on the master curves.

DeMartine and Cussler (1975) developed fluid mechanics-based models for predicting spreadabilty, viscosity and stickiness of skin cream. These were tested on silicone oils (2.6 - 85,000 mPa.s) and non-Newtonian fluids (aqueous solutions of hydroxypropyl methylcellulose and polyacrylamide) using a panel. Correlation coefficients of 0.9 or greater were found between predicted and subjective assessments. Results showed perception for attributes assessed using a shearing motion (spreadability and viscosity) was related to shear force with (approximately) constant velocity. For attributes assessed by normal motion (stickiness), the perception was proportional to time or velocity with (approximately) constant force. The attribute stickiness had the most complex relationship and predictions for stickiness were less successful in comparison to the subjective panel assessment. DeMartine & Cussler (1975) stressed that their results were limited as, although the apparent viscosity of the fluids varied widely, other physical properties likely to impact on the results such as fluid elasticity, fluid finger wetting and fluid thermal conductivity were not considered.

Wegener (1997) worked on materials with a wide range of viscosities suitable for skin cream application, including oils, o/w and w/o emulsions and gels. Samples were designed to include textures that are not currently observed in the personal care products market (they were not designed as personal care products). A trained sensory panel was used to objectively rate the samples and results were correlated to rheological parameters. The majority of sensory attributes measured were found to be closely related to the viscosity. The firmness and elasticity were related to the complex modulus, the visual creaminess and creamy feel were related to the phase angle and stringiness was related to the Arrhenius exponential index. It was reported that data from sensory assessments was most closely correlated to logarithmic values from rheological measurements. Predictive models predicting sensory properties from rheological parameters were developed showing good reliability ($R^2 > 0.75$) for the majority of attributes.

Although a broad range of research investigating the relationship between instrumental and sensory data is reported in the literature, it is clear that further research in this area would be beneficial. In particular, limited literature is available relating consumer liking to sensory properties of cream products. Instrumental and objective sensory data provide quantitative information about what the products are like (e.g. viscosity and skin feel) which is very useful in quality control and maintaining consistency between batches of skin creams. However, in order for a product to be successful, the consumer needs to like the product. Hedonic data enables the characteristics of samples that are desirable from a consumer's perspective to be identified. Therefore combining these fields of research provides valuable information for the new product development of successful cream products.

1.8 AIMS AND OBJECTIVES

The aims of this research were to:

1. Understand the relationship between sensory attributes and rheological parameters of skin creams.
2. Understand which product attributes are key drivers of consumer acceptability.

In order to achieve these goals, various sensory and physical methods were used to analyse the model skin creams that were produced according to an experimental design. These methods are outlined in Figure 1.5 in which arrows indicate how the methods link together.

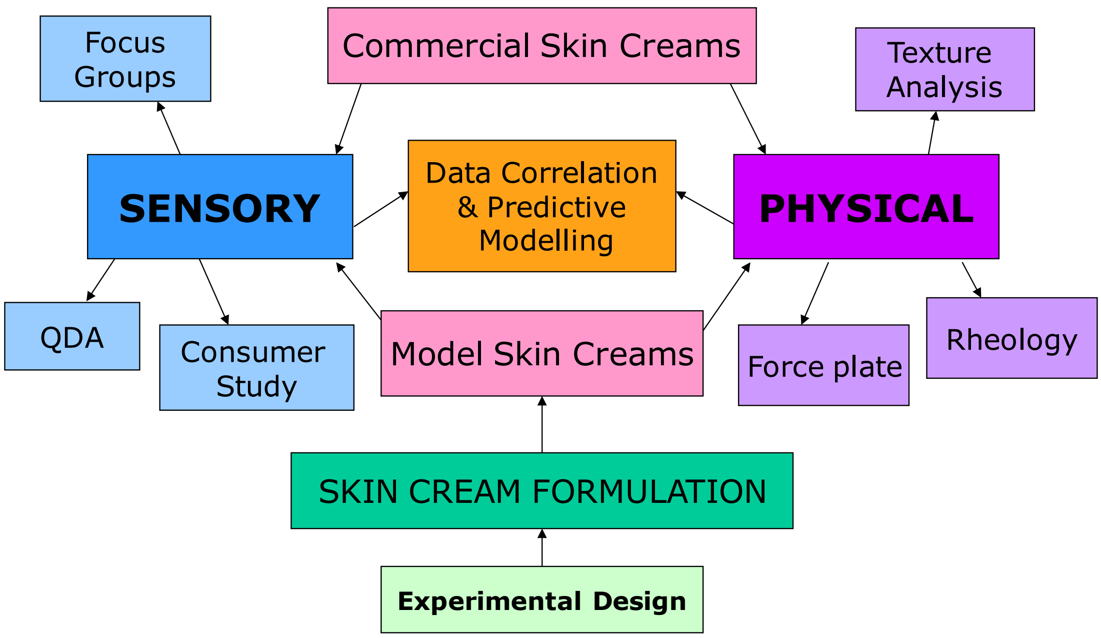

Figure 1.5: *Summary diagram illustrating how the different areas of research for this PhD link together*

Preliminary research was carried out on 8 texturally different commercial cream samples. These creams were characterised rheologically following various measurement protocols and using different geometries to determine the optimum test conditions for measuring creams with a wide range of textures. Focus groups were also carried out using these samples to observe application procedures consumers use when applying creams of differing textural properties.

For the main project work, model skin cream formulations were used. These were manufactured according to an experimental design (d-optimal response surface design, see Chapter 1.1.2), which allowed 40 samples with a wide range of textural properties to be produced from a minimal number of ingredient types and levels.

Quantitative descriptive analysis (QDA) was carried out whereby 10 trained panellists rated the model skin creams for 10 sensory attributes relating to the texture of skin creams. The creams were also characterised rheologically through oscillatory and steady shear measurements. The aim was to select measurements from which parameters could be extracted that related to the attributes measured by the trained panel. This would allow understanding of the relationship between sensory attributes and rheological parameters to be made (see Aim 1). Relationships between sensory attributes and rheological parameters were visualised using principal component analysis (PCA). The correlation matrix was used to identify any parameters that were highly correlated.

A subset of 12 model skin creams covering a wide range of textural properties was selected for use in a consumer study. These samples were freshly manufactured and hedonic data was collected as participants rated their like/dislike for each sample on a labelled affective magnitude (LAM) scale. Consumer study liking results were analysed in conjunction with sensory attribute data via cluster analysis and external preference mapping to determine which attributes were driving consumer liking behaviour (see Aim 2). The 12 creams were also rated by the trained panel for the 10 sensory attributes and measured instrumentally through rheological characterisation, texture analysis and force plate analysis. Relationships between sensory properties and physical parameters were visualised through PCA and the correlation matrix identified any highly correlated parameters.

Predictive models were generated from which sensory properties of skin creams could be predicted using physical data. These models could be used in conjunction with consumer study results to predict whether consumers would like creams with certain physical properties. The ability to predict the sensory properties a cream will have using instrumental data only is highly beneficial to manufacturers as the expensive, time consuming, sensory step is removed thus allowing more money, time and effort to be spent on developing new products.

In summary this PhD research attempts to correlate instrumental parameters to objective sensory data (trained panel attribute rating scores) so that models predicting sensory properties from instrumental data could be generated. Hedonic data was also collected in this research to enable an understanding of the desirable characteristics of skin creams to be gained, thus identifying which models would be more relevant in new product development.

2. MATERIALS AND METHODS

2.1 COMMERCIAL SKIN CREAMS

In order to identify attributes specific to different types of cream and to understand consumer opinions for products that already exist, eight commercial products (including hand and body creams and lotions) with a wide range of properties were purchased. These samples were used in focus groups where procedures by which consumers applied the creams and lotions to their hands were observed. Further information about these creams including composition, cost and special claims is given in Appendix I, Table A1.1.

2.2 MODEL SKIN CREAMS

2.2.1 Composition

The main research in this project was conducted on model skin creams that were designed to cover a wide range of textural attributes found in skin cream products. All creams were water continuous and composed of a limited number of hydrophobic and lipophilic ingredients in order to limit the complexity of the experimental design.

The **oil phase** contained oil - light **mineral oil** or **silicone oil** *(50 cSt and 20 cSt poly(dimethylsiloxane).* Note that creams containing silicone oil contained 50 % of the 50 cSt silicone oil and 50 % of the 20 cSt silicone oil as these levels resulted in an overall viscosity similar to that of the mineral oil used); **stearic acid (SA)** *(purum > 97 % GC)*; and 1-hexadecanol (**cetyl alcohol, CA)** *(purum > 95 % GC)* (all ex Sigma Aldrich, UK). Oils act as emollients to enhance skin feel (improve softness and smoothness, see Chapter 1.3.4) (Salka, 1997; Rawlings et al., 2004; Zocchi, 2009), the SA acts as a structuring agent giving body to the sample and the CA helps to stabilise the system (Eccleston, 1997; Telford, 2007). The **water phase** contained **RO** (reverse osmosis) **water** and **triethanolamine** (**TEA**) (> *99 % minimum)* (ex Sigma Aldrich, UK*).* **Thickener** was also added to the water phase in some formulations to improve stability, provide further body (Epstein, 2009) and enhance skin feel (Braun, 1991; Vanderbilt, 2004) thus adding variety to the overall texture of the skin creams. **Veegum** *(Magnesium Aluminium Silicate)* (ex R.T. Vanderbilt, US) and **Carbopol** *ULTREZ 10 NF* (ex Lubrizol, USA) were chosen for this purpose. Mixing the oil and water phases together allows the TEA and SA to interact causing neutralisation of the SA by TEA. This results in soap (triethanolamine stearate), which acts as an emulsifier improving the dispersion of oil within the formulation (Eccleston, 1997), see Figure 2.1.

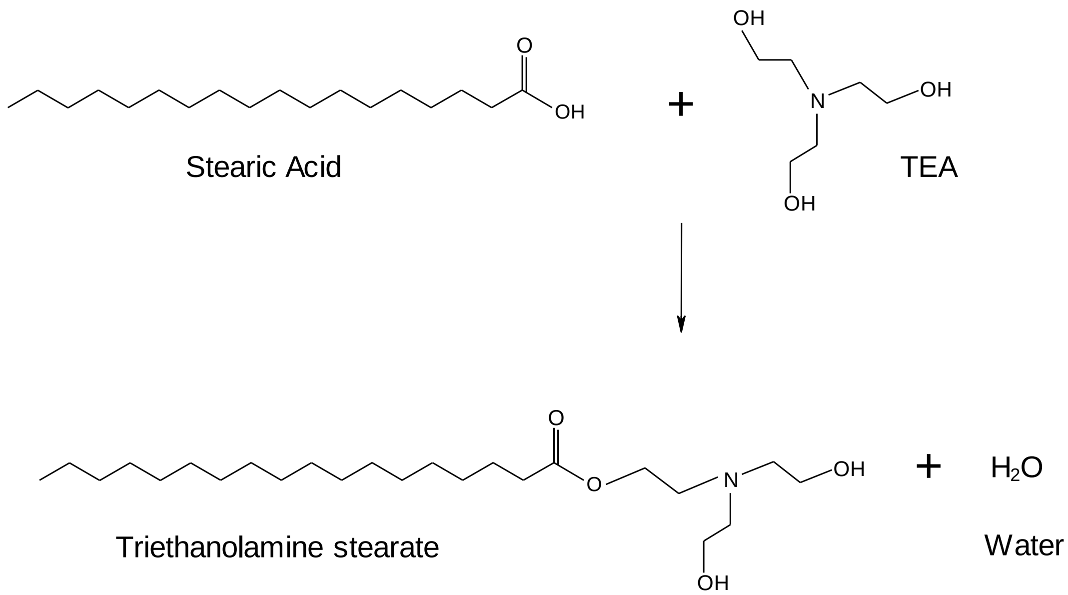

Figure 2.1: *Schematic showing the mechanism by which SA and TEA react to form triethanolamine stearate (the emulsifier) and water (Zhu et al., 2007).*

Guidance regarding suitable levels of ingredients for skin cream formulation was obtained from the literature (Forster and Herrington, 1997; Prinderre et al., 1998; Korhonen et al., 2001; Wibowo and Ng, 2001; Maccioni et al., 2002; Ribeiro et al., 2004; Moulai Mostefa et al., 2006) and through personal communication (Telford, 2007). Preliminary bench top skin cream manufacture revealed critical levels of stearic acid required in formulations - less than 2 % w/w resulted in unstable cream formulations. Ingredient types and levels causing the largest variation in sensory properties were also identified and incorporated into the final experimental design. Table 2.1 shows the range of ingredient concentrations applied throughout this project.

Table 2.1: *Concentration of ingredients used in model skin cream formulations. Values represent the percentage of the total composition on a weight-by-weight basis.*

Ingredient	MINIMUM (% w/w)	MAXIMUM (% w/w)
Mineral Oil	0	40
Silicone Oil	0	40
Stearic Acid	5	20
Cetyl Alcohol	1.25	1.25
Triethanolamine	0.5	5
Veegum (thickener)	0	1
Carbopol (thickener)	0	1
RO water	32.75	93.25

The different oil types were chosen to discover whether, for equivalent skin cream formulations, they impart different sensory or rheological properties. Likewise two types of thickener were chosen for use in this research due to their different thickening mechanisms which result in different sensory and rheological properties. Veegum is derived from clay; its particles are made up of thousands of submicroscopic platelets. Each platelet has a positive end and a negative face. The platelets are sandwiched together with a layer of water containing sodium ions to balance out the negative charge of the faces. On addition to water, the platelets are forced apart as water penetrates between the platelets. This separation of the platelets causes the positively charged ends to be attracted to the negatively charged faces forming a three dimensional colloidal structure, commonly referred to as a 'house of cards' type structure, (Vanderbilt, 2004).

Carbopol on the other hand is made up of cross-linked acrylic polymers (Barry and Meyer, 1979; Braun, 1991). On addition to water the polymer chains are in a tightly coiled state. However, in the presence of a base such as TEA, the carboxyl groups on the polymer chain are neutralised forming salt which allows the polymer chains to uncoil. Cross-links between the uncoiled chains then reform, thus providing maximum thickening efficiency (Braun, 1991; Gruber, 1999). The cross linked structures formed can be referred to as swollen microgels (Gruber, 1999). These different thickening mechanisms account for the different rheological and sensory properties. Carbopol forms much stronger bonds between polymer chains maintaining a stronger network structure, less easily deformed through stress, while Veegum is held together by the positive and negative attractive forces that are more easily overcome resulting in the weaker structure. Therefore for equivalent cream formulations, those containing Carbopol are likely to be thicker and more difficult to spread than those containing Veegum.

Having established suitable ingredients and their levels for the range of skin cream properties desired to be included in the core study of this research, the experimental design could be created. From a range of possible experimental designs, a D-optimal response surface design was considered most suitable for this application since it selects the minimum number of samples required to obtain accurate models (see Chapter 1.1.2). In this case 40 samples were identified, which was substantial but manageable for the time frame allocated to this PhD.

D-optimal designs select strategic samples including replicates and lack of fit points (Eriksson et al., 2000). The latter, together with standard design points, enable accurate predictive models to be made. Standard design points ensure that good ranges of model terms are included within the design so accurate predictions can be made. Replicate design points allow estimates of the experimental error to be calculated. Lack of fit points are located at different places to the standard design points within the design space, this allows lack of fit statistics to be calculated in which experimental and residual error[1] are compared. Significant lack of fit suggests the model is inaccurate and should not be used to make predictions (Design-Expert, 2000).

The experimental design is given in Table 2.2 where columns illustrate the types and levels of ingredients required in each formulation. The sample 'column' provides the numbers by which creams are referred to throughout this thesis. It should be noted that there are seven replicate compositions within the experimental design indicated by 'R'. Replicate samples were selected by the computer software as points within the design with the highest leverage i.e. the highest influence on the overall design (Design-Expert, 2000). Note that some of the cream samples in the design contained no oil. An example of a commercial cream containing no oil is Neutrogena concentrated hand cream (see Table A1.1, Appendix I), this type of cream could be classed as a vanishing cream due to the high level of water and lack of oil which will prevent a greasy residue forming on application (see also Chapter 1.3.4).

[1] Residual error – the difference between the observed response and the value predicted by the model for a particular design point
Experimental error – the normal variation in the response as observed when an experiment is repeated from scratch *(Design Expert, 2000)*

Table 2.2: *Experimental design for model skin cream formulations where all samples contained 1.25 % CA, and RO water made the remaining percentage up to 100 %. Creams used in the consumer study are highlighted while those used in practice rating are indicated by *.*

	Factor 1	Factor 2	Factor 3	Factor 4	Factor 5	Factor 6
Sample	**Oil**	**Stearic Acid**	**TEA**	**Thickener**	**Oil Type**	**Thickener Type**
	% w/w	% w/w	% w/w	% w/w		
1	0	20	0.5	1	Mineral	Veegum
2	0	5	5	0	Silicone	Veegum
3*	0	12.5	5	0	Mineral	Carbopol
3R	0	12.5	5	0	Mineral	Carbopol
4	40	5	5	0.5	Mineral	Carbopol
4R	40	5	5	0.5	Mineral	Carbopol
5*	40	20	5	1	Silicone	Carbopol
5R	40	20	5	1	Silicone	Carbopol
6	0	5	0.5	0	Mineral	Carbopol
7	20	12.5	2.75	0.5	Mineral	Veegum
7R	20	12.5	2.75	0.5	Mineral	Veegum
8	0	5	0.5	1	Silicone	Veegum
8R	0	5	0.5	1	Silicone	Veegum
9	40	20	0.5	1	Silicone	Veegum
10	0	5	2.75	0	Mineral	Veegum
11	20	5	5	0	Silicone	Carbopol
11R*	20	5	5	0	Silicone	Carbopol
12*	0	20	5	0	Mineral	Veegum
12R	0	20	5	0	Silicone	Carbopol
14	40	20	0.5	1	Mineral	Carbopol
15	20	12.5	2.75	0.5	Mineral	Carbopol
16*	40	5	2.75	1	Silicone	Carbopol
17	40	20	5	0	Mineral	Veegum
18*	20	12.5	2.75	0.5	Silicone	Veegum
20*	0	20	5	1	Silicone	Veegum
23	40	20	2.75	0	Silicone	Carbopol
24	20	12.5	2.75	0.5	Silicone	Carbopol
25	40	5	5	0	Silicone	Veegum
27	0	20	5	1	Mineral	Carbopol
28	40	5	0.5	0	Mineral	Veegum
29	0	12.5	0.5	0	Silicone	Veegum
30	0	20	0.5	1	Silicone	Carbopol
31	0	5	5	1	Mineral	Veegum
32	40	20	0.5	0	Mineral	Veegum
33	0	5	0.5	1	Mineral	Carbopol
34	0	20	0.5	0	Mineral	Carbopol
35	40	5	0.5	0	Silicone	Carbopol
36*	40	5	0.5	1	Mineral	Veegum
37	0	5	5	0.5	Silicone	Carbopol
40	40	20	5	1	Mineral	Veegum

2.2.2 Manufacture of model skin creams

Skin creams were produced in 3 kg batches using the skin cream rig depicted in Figure 2.2 (on loan from Unilever, the industrial sponsor of this project). The rig consisted of a large jacketed vessel (approx. 21.5 cm diameter at the widest point, 21 cm depth at the deepest point, 5 kg capacity), a wall scraping paddle stirrer (paddle height 13.5 cm; maximum width approx. 19.5 cm), motor for the stirrer (Heidolph, RZR 2021, 40-2000 rpm range) and a hot water bath (HWB; Haake L water bath with Haake D8 Heating Circulator) attached to the jacketed vessel through 3-way valves.

The method of skin cream production follows: Initially, water was added to the jacketed vessel and heated to 70 °C (HWB set to 83 °C) whilst stirring at 70 rpm. The oil, SA and CA were added and mixed at 100 rpm until a homogenous mixture was obtained. Maximum shear rates in the order of 50 s^{-1} were generated. The HWB was switched off and the TEA added. After 10 minutes, the rotor speed was reduced to 70 rpm and cooling continued until the sample reached 50 °C (after 1 - 2 hours) at which point thickener was added and the mixture was left for 1 hour to mix at 50 rpm. Once samples had cooled to ≤ 30 °C, they were removed from the jacketed vessel and transferred to the mixing bowl of a domestic mixer (Kenwood Chef, KM001, 1000 W, 4.6 l capacity) and mixed for approximately 3 minutes (setting 4) using a beater (K-beater, stainless steel). Subsequently, the samples were transferred to a refrigerated room kept at 4 °C. After 24 hours, creams were mixed once again for 3 minutes (domestic mixer, setting 3).

This apparently complex protocol for cream manufacture was the result of preliminary trials, which resulted in creams of inhomogeneous consistency and microstructure instability such as creaming or separating out of the oil phase. The protocol described allowed manufacture of stable skin cream samples. The temperatures were chosen to ensure that on addition of the oil phase the SA and CA would melt as the melting point of SA is 69 °C (Sigma-Aldrich, 2006) and that of CA 49 – 50 °C (Sigma-Aldrich, 2006). Note that the TEA and thickener were diluted in a portion of water from the water phase to allow for more efficient transfer to the jacketed vessel and improved mixing on addition to the formulation.

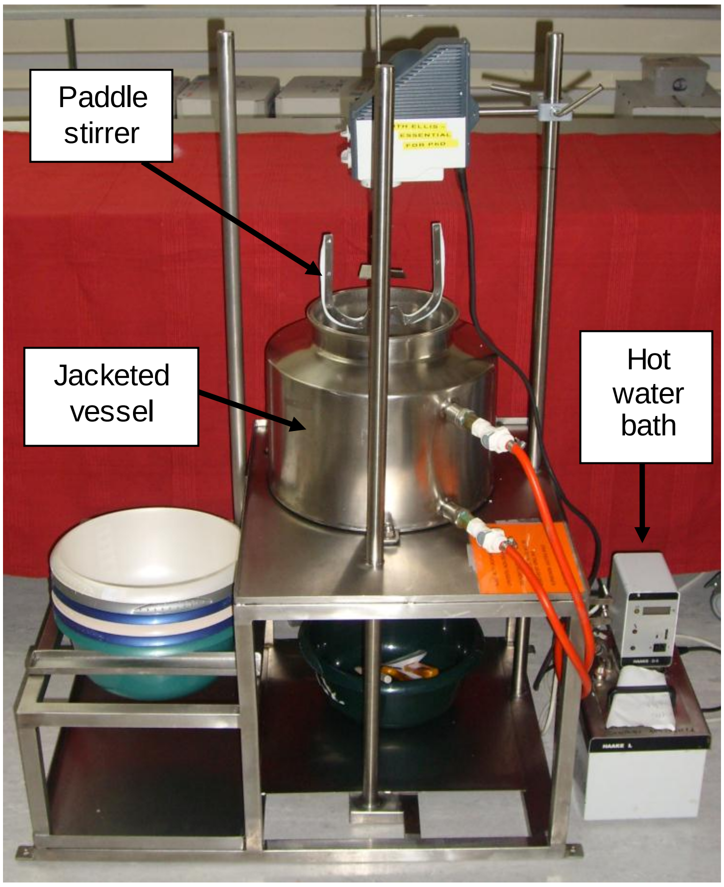

Figure 2.2: *Skin cream rig (5 kg capacity).*

Although no colour or perfumes were added to the samples, the overall visual appearance of the skin creams differed. Creams containing veegum had a beige tinge and the thickness of a cream could also be inferred visually. No attempts were made to disguise these factors so the appearance could have affected the overall sensory perception. Some formulations contained air bubbles, in particular thinner samples, which may have affected results from physical measurements.

2.3 SENSORY METHODS

The main sensory objectives of this PhD were to identify key textural attributes present in skin creams and subsequently to quantify these using a trained panel. Results could then be used to determine which attributes were driving consumer liking.

In Chapter 2.3.1 the use of focus groups to understand consumer application behaviour is described. In Chapter 2.3.2, the sensory methods involved in the QDA study on the skin creams are detailed. Sensory attribute protocols derived from QDA for rating the 40 model skin creams are also given. Preliminary results from training sessions are mentioned where findings were relevant to the final study. Statistical methods used for analysis of QDA results are also described. Chapter 2.3.3 provides an overview of the methods used in the consumer study including: the selection of a subset of 12 model skin creams, procedures employed to collect the hedonic data and the methods used to analyse the results including standard preference mapping and an approach looking at consumer liking in terms of satisfaction and dissatisfaction scores.

2.3.1 Understanding consumer application behaviour of skin creams

Focus groups were carried out using commercial skin creams (see section 2.1) in an attempt to understand application procedures used by consumers when judging how much they like a skin cream sample. Two focus groups were held involving eight and ten participants respectively. Application procedures most commonly used by the recruited consumers are given in Appendix II, Table A2.1. In summary, results showed that individuals have their own preferred way of applying skin cream, as formed by habit. The general application procedure followed by an individual was similar for a range of products however textural attributes such as thickness affected the way subjects applied skin creams. For example creams of low viscosity tended to be applied at a faster shear rate than thicker creams. Brummer and Godersky (1999) also found that thinner samples (lotions) were applied at a faster shear rate than thicker samples (creams), see Chapter 1.8.

Characteristics of creams liked by individuals varied depending on skin type and personal perception i.e. the feel of the cream on the skin. Brand imaging also had a major impact on whether individuals liked or disliked certain creams. For example ‘Mango Body Butter’ was considered to be a luxury product and received much praise from the consumers whereas ‘Boots Basics hand and body lotion’ was considered undesirable and obviously ‘cheap’.

Background information gained from focus groups was invaluable for advising the University of Nottingham (UoN) external sensory panel when developing skin cream application protocols for use during the quantitative descriptive analysis (QDA) of the model skin creams. In particular the application procedures observed (see Appendix II, Table A2.1) whilst consumers made references to the texture and skin feel of the products was useful in understanding which attributes are important to consumers and how they judge such attribute properties.

2.3.2 Measuring textural characteristics of skin creams

2.3.2.1 Panellists

Ten panellists (eight female, two male; aged 43 - 70) from the UoN external sensory panel were invited to take part in this study after completing appropriate screening tests with commercial skin cream samples (23 panellists were screened in total). Panel screening involved a series of basic rank rating tests on the commercial skin creams to determine which panellists, with little practice, were good at rating textural properties of skin creams. All panellists had been members of the UoN sensory panel for between five and ten years and had experience of a wide range of products. Although this was the first time the sensory panel had carried out work on skin creams, they had previous experience of the general methods of sensory profiling used in this project.

Quantitative descriptive analysis (QDA) of the 40 model creams (including training and rating sessions) took place over nine months in which panellists attended two to three sessions per week, each lasting approximately two hours. Ethics approval was granted by the UoN Medical Ethics Committee and all participants filled in forms to give their consent to be involved in the study.

2.3.2.2 Attribute generation and definition

In individual booths, panellists were asked to write down all the textural attributes (both visual and tactile texture) they could think of to describe the different model skin creams. Samples were presented to panellists in a random order one at a time in 25 mL lidded, clear containers each labelled with a 3-digit code to identify the sample. The random order was determined by use of a random order table (Meilgaard et al., 1999).

Terms ('descriptors') generated to describe the different textural properties of the creams were combined on flip charts as panellists reconvened in a training room for discussion. An example of the descriptors generated in one session is given in Figure 2.3. The tally lines next to the words indicate the number of panellists who used that term to describe the skin creams. Three digit codes next to these terms refer to the samples that the panellists agreed possessed that attribute. The arrows link up words that through discussion the panellists agreed have similar meaning. This is a very important part of descriptive profiling. For example, one panellist used the word 'cloying'. To other panellists this word was meaningless but through discussion it was revealed that this panellist was using the term 'cloying' to describe what others had labelled 'stickiness'.

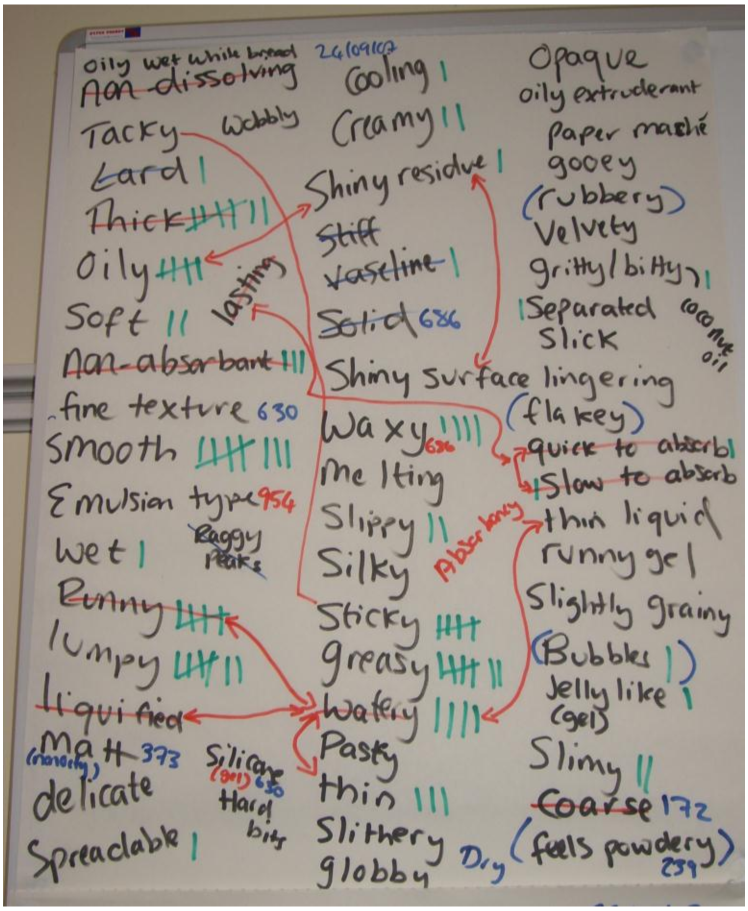

Figure 2.3: *Attributes generated by descriptive profiling panel session 1 using creams 2, 4R, 6, 11, 16, 25, 27, 40 (see Table 2.2 for composition).*

Rationalisation and definition of terms was conducted in a training room where each panellist was given cream samples in 25 mL, lidded pots (eight samples were discussed per session) and finger bowls containing water with lemon juice were provided. Reference materials were used to aid panellists in describing and understanding the properties of the cream. These were vegetable oil (Sainsbury's Vegetable Oil) for oiliness, butter[1] (Lurpak Spreadable) and margarine[2] (Stork) for greasiness, a jelly cube[3] (Sainsbury's Strawberry Flavour Jelly) and hair gel (Coop

[1] Ingredients: Butter, Vegetable Oil, Lactic Culture, Salt (0.9%)

[2] Ingredients: Vegetable Oils, Water, Salt (1.75%), Buttermilk, Emulsifiers: Mono- And Di-glycerides of fatty acids, Flavourings, Vit E, Citric Acid, Preservative (Potassium Sorbate), Colour: Beta-Carotene, Vitamins A & D.

[3] Ingredients: Glucose-Fructose-Syrup, Sugar Solution (Sugar,Water), Pork Gelatine, Citric Acid, Acidity Regulator: Sodium Citrate; Natural Flavouring, Colour: Anthocyanins, Curcumin; Acetic Acid.

gel, extra firm hold[1]) for gel-like. In total, five sessions were held in which the initial attribute terms were discussed to ensure there was agreement on the selected definitions and any confusion over terms was identified.

2.3.2.3 Attributes selected for rating model skin creams

Following extensive discussion, panellists selected 11 attributes that they felt were suitable to measure the range of textural properties encompassed by the 40 model skin creams:

- Firmness
- Thickness
- Resistance
- Spreadability
- Stickiness
- Cooling
- Drying
- Dragging
- Slipperiness
- Absorption
- Final greasiness

The panel composed detailed reference protocols with definitions for these attributes to ensure everyone followed the same procedure when rating in the booths. These definitions are given in Table 2.3. The order given in Table 2.3 was selected so that the process of rating one sample for the 11 attributes could be carried out in one sitting without hand washing.

[1] Ingredients: Water, Triethanolamine, Carbomer, PVP, Polysorbate, 20, Sodium Methylparaben, DMDM Hydantoin, Disodium EDTA, Panthenol, Colours (CI 17200, CI 42051), Perfume (contains linalool)

Table 2.3: *Textural attribute definitions as supplied to the external panel for reference throughout rating of model skin creams.*

The following four attributes are rated one after the other.

Attribute	Definition	Anchors
	Gently dip <u>index finger</u> in cream, observe resistance of cream to movement. Assess:	
Firmness	Overall firmness of product, ranging from:	not to very

	Dip <u>index finger</u> in cream, and press to side of pot. Assess:	
Thickness	Overall thickness of product, ranging from:	not to very

	Dip <u>index finger</u> in cream, rub between thumb and Index finger five times. Assess:	
Resistance	How easy it is to move thumb and index finger from:	easy to difficult

	Use remaining cream on <u>index finger</u> (from resistance test). Spread sample on <u>back</u> of hand with index finger, from wrist to knuckle back and forth twice. Assess:	
Spreadability	How easy it is to spread the cream over back of hand from:	easy to difficult

NOW RINSE YOUR FINGERS AND BACK OF HAND USING THE FINGER BOWL PROVIDED THEN RATE THE NEXT ATTRIBUTE

	Use spatula to get a spoon full of cream, scrape spatula against edge of pot until level. Use index finger to remove cream from spatula. Spread sample on back of hand from wrist to knuckle once Press and release <u>middle finger</u> on back of hands 3 times Assess:	
Stickiness	Overall stickiness of product, ranging from:	not to very

NOW RINSE YOUR FINGERS AND THE BACK OF YOUR HAND

Attribute	Definition	**Anchors**
	Use spatula to get a spoon full of cream, scrape spatula against edge of pot until level. Use index finger to remove cream from spatula. Wipe cream onto inner side of wrist and leave for 15 seconds. Assess:	
Cooling	Overall cooling feel of product, ranging from:	not to very

The following two attributes are to be rated one after the other <u>i.e. in one application of cream</u>

	Use spatula to get a spoon full of cream, scrape spatula against edge of pot until level. Use index finger to remove cream from spatula. Rub sample rapidly over back of hand using 3 fingers for 20 seconds Assess:	
Drying/Taut feel	How drying or taut the skin feels following application from:	not to very

	Directly after rating drying feel….. Run different finger, over the area of skin to which sample was applied Assess:	
Dragginess/ Rubbery	How much the skin drags as fingers move over the skin once cream is absorbed from:	not to very

NOW RINSE YOUR FINGERS AND BACK OF HAND USING THE FINGER BOWL PROVIDED THEN RATE THE NEXT ATTRIBUTE

The following three attributes are to be rated one after the other <u>i.e. in one application of cream</u>

	Use spatula to get a spoon full of cream, scrape spatula against edge of pot until level. Use index finger to remove cream from spatula. Rub sample on back of hand in circular motion for 5 seconds. Assess:	
Slipperiness	Initial slipperiness of cream, i.e. how well cream slides on skin from:	not to very

	Directly after rating slipperiness, continue rubbing the sample in for a further 25 seconds. Assess:	
Absorption	How quickly the cream is absorbed from:	slow to fast

	Directly after rating absorption, run a different finger over area of skin to which cream was applied. Assess:	
Final greasiness	Final greasiness feel on skin from:	not to very

2.3.2.4 Procedure for rating model skin creams

Rating model skin creams for the previously defined attributes was achieved using continuous line scales designed in Fizz data acquisition and analysis software (Fizz, version 2.20C, 1994-2006, Biosystems, France), see Figure 2.4. The ends of the scale represented 0 (‘not’) to 100 % (‘very’). Panellists were encouraged to use the full range of the scale based on the product space encountered during training. They were informed that the model cream samples represented the whole range of the line scale therefore the thickest sample should be rated at the 100 % end of the scale, next to the ‘very’ anchor.

Figure 2.4: *Example of a line scale used for rating attributes of skin creams.*

In individual booths, panellists followed the rating protocols given in Table 2.3 for each cream sample as presented to panellists separately in 25mL containers. Panellists were provided with a finger bowl containing water and lemon juice to remove greasy residue from fingers between rating the different attributes and kitchen roll was provided for hand drying (see Figure 2.5 for booth set up). This procedure ensured minimal disruption to the rating process. Panellists washed their hands with soap[1] (in a standard kitchen sink) between samples to reduce carry-over effects. A 10-minute break was also taken before rating the next sample to allow for skin recovery.

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[1] Liquid Soap, SCA Hygiene products SE-405 03 Göteborg Sweden.
Ingredients: *Aqua, Sodium cocoamphacetate, Lauric acid, PEG-4, Rapeseedamide, Myristic acid, Potassium hydroxide, Glycol stearate, Potassium carbonate, Methylparaben, Propylparaben, Lactic acid*

Timers were supplied in the booths so panellists could monitor their application procedure and therefore be as consistent as possible in their rating technique. A stainless steel spatula *(Fisher-Scientific, 10 μL volume)*, see Figure 2.6, was also provided to each panellist to ensure that the same sample size of cream was used when rating specific attributes *(see Table 2.3).*

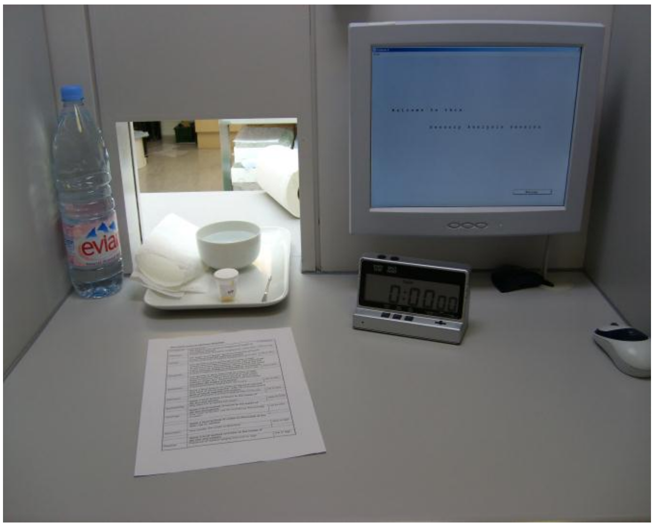


Figure 2.5: *Example of booth set up for rating samples.*

Figure 2.6: *Spatula used by panel to improve consistency when rating certain attributes (Fisher-Scientific size 8 x 6 x 1.5 mm, 150 mm length, 10 μL volume).*

2.3.2.5 Training to measure the attributes

Training sessions involved both standard rank rating and rating techniques to improve the panellists overall discrimination ability. These sessions highlighted areas where the panel needed re-training and gave panellists a better idea of the challenging attributes. For example, panellists originally rated 'initial greasiness'. The results, however, were not consistent amongst panellists. Through discussion, it was then decided that the term 'initial greasiness' should be changed to ‘slipperiness’ as this was in fact what the panel were rating. This new term was understood far better by the panel and results improved. Figures A3.1 and A3.2 in Appendix III show results obtained when rating initial greasiness and slipperiness, respectively. A high level of cross over can be seen in Figure A3.1, which suggests panellists were rating the samples in different ways to each other. On the other hand, Figure A3.2 shows greatly improved results where a clear pattern is observed.

Panellists were reminded to be consistent between sessions and to try to follow the rating protocol as strictly as possible to minimise the chance of errors. The following key factors that affected their results were highlighted: 1) Sample size used; 2) Method of application – speed of rubbing, area over which sample is applied, pressure of finger on sample; 3) Temperature of hand, finger and wrist; and 4) Washing hands.

Based on the panel’s comments during training and results from rating samples in the training room, eight skin creams covering a range of the identified attribute properties, were selected for practice rating in the sensory booths. These were creams 3, 5, 11R, 12, 16, 18, 20 & 36, see Table 2.2 for composition. Panellists attended four sessions; six samples were rated per session for the 11 attributes, resulting in triplicate data for each panellist for the eight samples. This preliminary subset was used to determine whether the panel was ready to start rating the full set of 40 samples. Fizz data acquisition and analysis software (Fizz, version 2.20C, 1994-2006, Biosystems, France) was used for data analysis along with SPSS statistical analysis software (SPSS 16.0 for Windows, SPSS Inc., 1989-2007).

Interaction graphs from preliminary rating of skin creams showed panellists were rating the different creams in a similar manner. One way ANOVAs highlighted panellists that were not discriminating between samples for the different textural attributes (see Table A4.1, Appendix IV). It was clear from these results that panellist J struggled to discriminate between the samples for the majority of the attributes ($p > 0.05$). This is likely to be due to the fact that this panellist was involved in a car accident the week before rating these samples, which resulted in the left wrist being in plaster for six weeks. Therefore this panellist had to get used to using the other hand for rating. The ANOVA results showed that the attribute cooling appeared to be more challenging than the others, where six out of ten judges were not discriminating between samples ($p > 0.05$, see Table A4.1, Appendix IV).

Tukey's Honestly Significant Difference (HSD) results showed that for most attributes, individual panellists were able to separate samples into two to five homogeneous subsets (out of a maximum of eight). However, for more challenging attributes (stickiness, cooling, drying and dragging) some panellists used only one subset indicating a lack of discrimination ability. Table A4.2, Appendix IV provides the number of homogeneous subsets individual panellists separated creams into for each attribute.

Two-way ANOVA showed significant panellist and panellist-product interactions ($p < 0.05$). Although the eight creams were selected to encompass the range of textural attributes covered by the 40 model skin creams, some of the samples were similar to each other for specific attributes and this could lead to the cross-over interactions causing the significant panellist-product interaction. Considering results obtained from the one-way ANOVA, these cross-over effects were deemed acceptable for the panel to rate the 40 model skin creams.

Overall preliminary rating highlighted attributes that were causing problems amongst panellists (cooling, drying, dragging and stickiness). It was therefore decided that during rating the 40 samples in triplicate, reference samples for these attributes, as well as thickness and final greasiness, should be provided prior to every fourth session as a reminder of the extreme samples. It should be noted that reference samples were <u>not allowed</u> in sensory booths, they were just available at the start of relevant sessions for reference. This prevented constant comparison in the booths which could lead to fatigue and potentially biased results.

2.3.2.6 Evaluation of model skin creams

Model skin creams were rated for the 11 attributes in triplicate. Booth and skin cream temperatures were recorded during each sensory session for reference purposes in case anomalous results were observed. Skin creams were removed from the refrigerator 1 hour 45 minutes prior to each sensory session; however, the average temperature of the cream during and between sessions varied due to the nature of the creams and the fluctuations in the temperature of the sensory kitchen respectively. Averaged over all assessments the cream temperature was 20 °C ± 5 °C and booth temperature 22 °C ± 3 °C.

Prior to each session, a digital thermocouple with a flat probe was used to record the temperature on the back of the panellists' hands (see Figure 2.7) and panellists assigned a rating to their skin condition using the following scale: 1 = very dry; 4 = normal; 7 = hydrated.

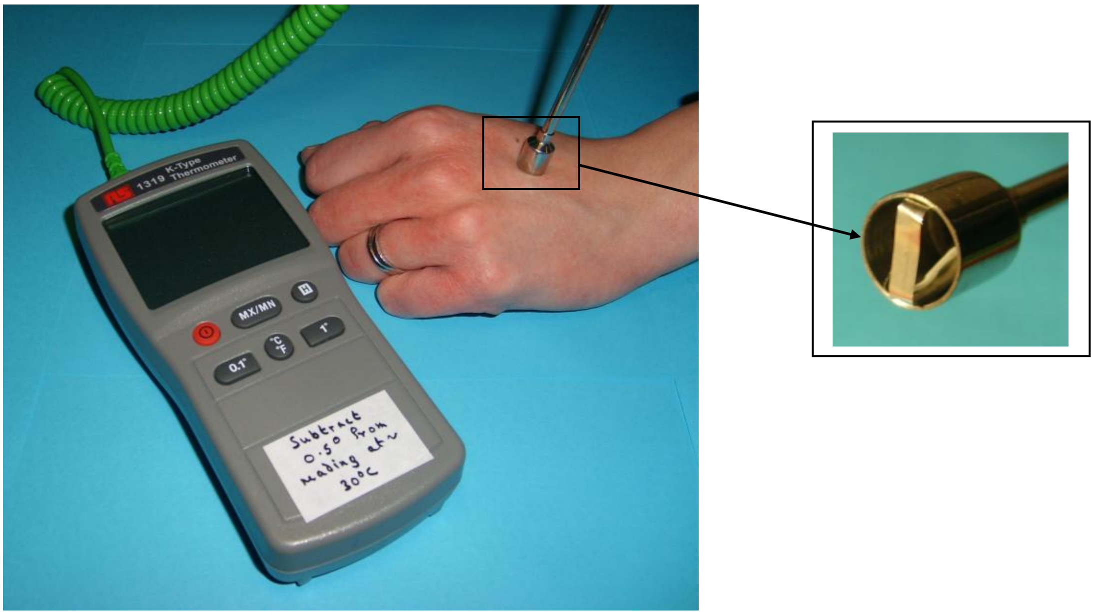


Figure 2.7: *Thermocouple with a flat probe to measure temperature on the back of panellists' hands.*

This data was useful as skin condition affects the way skin cream is perceived by the panellists as was observed in earlier training sessions. For example, the hotter the skin, the easier the cream was found to spread (see also Chapter 1.3.2). Also, the properties of dry skin vary considerably to normal or hydrated skin in their ability to hold moisture (Rudikoff, 1998; Rawlings and Matts, 2005; Couturaud, 2009), therefore it is likely that skin hydration may affect rating of some attributes in particular those involving absorption into the skin (drying, dragging, absorption and final greasiness). Relatively consistent results were obtained for skin condition although for some panellists their hand temperature varied greatly between sessions. The largest temperature difference observed between sessions was 9.4 ºC (panellist J) although in most cases differences between sessions were smaller (± 3 ºC).

Panellists attended 21 ratings sessions in which the 40 model skin creams were rated in triplicate (15 sessions rating 6 samples; 6 sessions rating 5 samples). A Williams Latin Squares Design as created in Fizz software was used to determine sample order for each session. Samples were rated in blocks therefore all panellists rated the same six creams per session although they were presented to them in a different order. Replicate samples were rated in different sessions. In each session, samples were rated for all 11 attributes (see Table 2.3) following the procedure outlined in Chapter 2.3.2.4.

2.3.2.7 Rating 40 model skin creams in triplicate: Absorption

Whilst rating skin creams for the 11 attributes it became clear that panellists were struggling greatly with the term Absorption. It was decided that this attribute should be rated in isolation. Therefore, following rating of 40 samples in triplicate, panellists attended a further four sessions where they rated all samples for the attribute absorption in triplicate; results were greatly improved with this method. The protocol for rating absorption in isolation is given in Figure 2.8.

<u>Absorption: METHOD</u>

- Use spatula to get a spoon full of cream
- Scrape spatula against edge of pot until level (i.e. just the 'bowl' at end of spatula is filled with cream).
- Use index finger to remove cream from spatula.
- Gently rub cream on BACK OF HAND, back and forth (from wrist to knuckle) with index finger for 30 seconds

Assess: How quickly the cream is absorbed from slow to fast on the line scale.

A cream has fully absorbed when you can no longer see the cream in its original state and when further rubbing no longer has an effect.

Figure 2.8: *Modified application protocol as used by the trained panel for rating the attribute absorption in isolation.*

2.3.2.8 Statistical analysis of descriptive profiling data

A range of statistical tests were applied to determine significant differences between the samples for the textural attributes investigated and to check panellist performance (SPSS 16.0 for Windows, SPSS Inc., 1989-2007).

Two – way ANOVAs (analysis by attribute with product and panellist factors) were performed on panel mean data to highlight significant differences between samples for each of the assessed attributes.

Tukey's Honestly Significant Difference (HSD) multiple comparison tests with interaction plots were used to determine how well panellists were discriminating between creams for the different attributes and allowed any differences between panellists in use of the rating scale to be observed.

Principal component analysis (PCA, XLSTAT, version 2007.6) was used to determine the relationship between the textural attributes and to highlight the differences between the model skin cream samples by grouping them in relation to these attributes (see also Chapter 1.4.3.1).

2.3.3 Consumer study

2.3.3.1 Samples

A subset of 12 skin cream samples (creams 1, 3, 5, 8, 9, 11R, 12R, 16, 18, 27, 28 & 32) from the 40 model skin creams was selected based on PCA results from descriptive profiling (see Figure 3.3, Chapter 3.1.2). These were creams from all four quadrants of the PCA biplot and some from the centre to ensure that the complete range of textural properties were included in the consumer study. These creams were freshly manufactured using the production methods described in Chapter 2.2.2.

2.3.3.2 Rating consumer study creams in triplicate

After initial re-training sessions, the trained panellists as selected from the UoN external sensory panel attended a further seven sessions in which they rated the 12 creams in triplicate according to methods described in Chapter 2.3.2.6. Rating of the consumer study creams was carried out to check batch to batch consistency in the creams and to allow predictive models to be made, see Chapter 4.2.

During re-training the application protocol for the attribute ‘absorption’ (see Figure 2.8) was modified slightly. Instead of rating absorption on a scale from slow to fast, panellists decided it was better to rate absorption from fast to slow since they were effectively timing how long it takes for the sample to absorb within the 30 seconds allocated to absorption rating. Fast absorption therefore related better to the ‘not’ side of the scale (see Figure 2.4) as very fast absorption would be closer to 0 seconds. Slow absorption on the other hand related to a longer time (closer to or exceeding the 30 seconds) hence it was better placed at the ‘very’ end of the scale. This improved the overall results see Figures A5.1 & A5.2, Appendix V.

2.3.3.3 Consumers

Consumers were invited to take part in this study based on their skin cream usage, availability, willingness to take part and lack of skin allergies as determined by a brief pre-screening questionnaire (see Appendix VI). A total of 150 naïve consumers were recruited (aged 16 - 60+). The UoN Medical Ethics Committee granted ethics approval and all participants filled in forms to give their consent to be involved in the study.

2.3.3.4 Collecting hedonic data

In consumer studies involving rating of a sequence of products, participants tend to score the first sample they receive higher than the rest (MacFie, 2007). In order to prevent such effects tainting the results of this study, data obtained for the first sample in each session was discarded. These samples are referred to as 'dummy' samples (MacFie, 2007) but participants were unaware of this procedure therefore they rated them as normal. Note that the dummy samples used in this study were additional to the 12 consumer study creams so consumers rated a total of 14 samples (including two dummy samples). Therefore liking data for all 12 creams was still obtained for each participant. Cream 16 was chosen for use as the dummy sample due to its medium thickness and appearance close to a standard cream one might buy.

Participants attended two sessions rating seven samples per session. They were asked to sample skin creams on the back of their hand using their index finger rating a maximum of two samples per hand. A finger bowl containing water and lemon juice was provided for finger rinsing between samples and kitchen roll was provided for hand drying. Once four samples had been rated, participants were asked to wash their hands thoroughly and have a ten minute break to allow for skin recovery prior to further rating.

Prior to each session, participants were reminded that samples involved in the consumer study were a 'hand cream base' only and therefore they may not be the 'most desirable' creams they have ever tried (no added colour or perfume). They were asked to focus on the function of the samples in particular on how much they liked or disliked the feel of the samples rather than their appearance. Note that the term 'hand cream' was used in the consumer study to ensure all participants judged the samples in relation to one type of cream. Liking is based on functionality (Brummer, 2006) so if the term skin cream had been used, results may have been confused with some consumers rating liking on the basis of it being a body cream while others may have focused on its appeal as a hand or face cream.

Testing took place in sensory booths where consumers received samples one at a time. The rating was computer aided, see Figure 2.9.

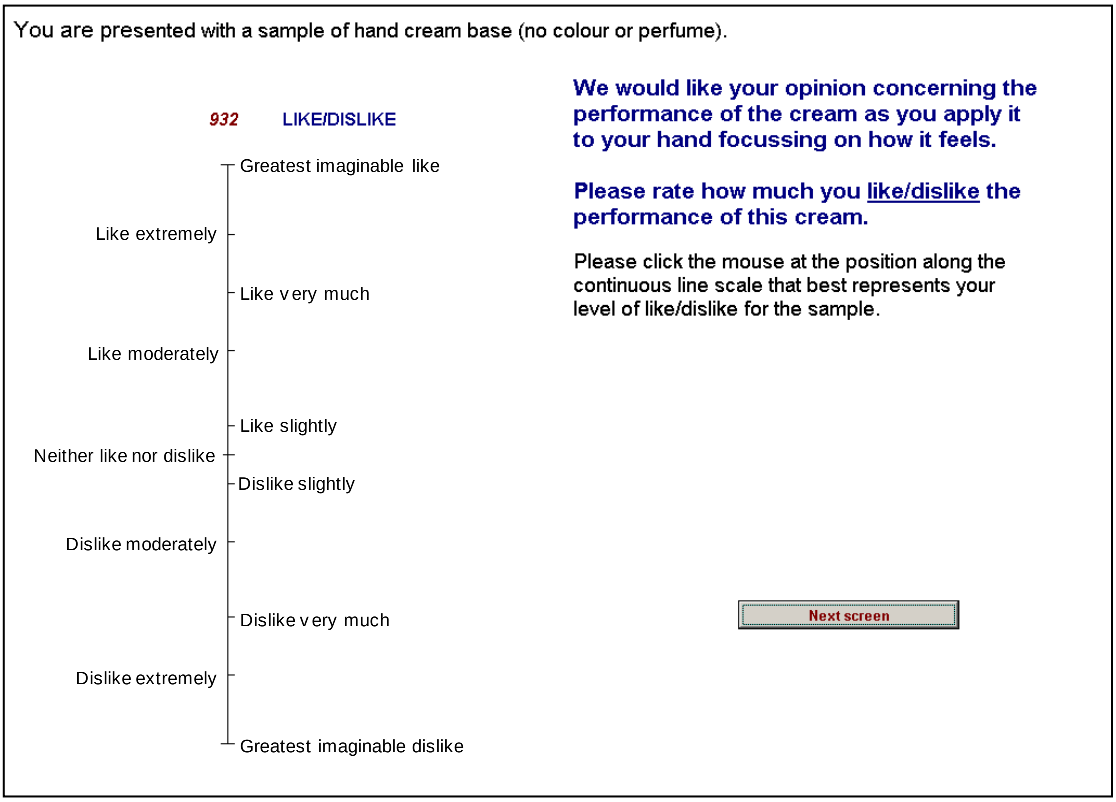


Figure 2.9: *Screen presented to consumers for rating liking of different hand cream samples.*

A LAM (labelled affective magnitude) as developed by Schutz and Cardello (2001) was selected for use in this study. The distance between labels on the LAM scale have been reported to be more reliable than those on a 9-point scale where divisions between anchors are equal and therefore unrepresentative of the magnitude of difference between them, thus the LAM scale provides better discriminative sensitivity than the commonly used 9-point hedonic scale (Schutz and Cardello, 2001; Greene et al., 2006; Nasser El Dine and Olabi, 2009). On the other hand, some literature reports no significant improvement in data results with the LAM scale compared to best-worst scaling (Jaeger and Cardello, 2009), the 9-point scale (Lawless et al., 2010) or an unstructured hedonic line marking scale (Hein et al., 2008). Despite this, it was decided that the LAM scale would still be a more appropriate choice. Since the creams used in this study were not commercial products, it was thought that liking would be limited. A 9-point scale would therefore restrict the participant's hedonic data whereas the LAM scale would enable more discrimination between different consumer liking behaviours.

2.3.3.5 Data analysis

Agglomerative hierarchical clustering (AHC or cluster analysis) and external preference mapping were carried out to enable a greater understanding of the relationship between the hedonic data from the consumer study and the textural attributes determined by QDA to be gained; thus attributes that were desirable from a consumer perspective could be identified. Background information about cluster analysis and preference mapping is given in Chapters 1.4.3.2 and 1.4.3.3 respectively.

Initially agglomerative hierarchical clustering (AHC) was used to reveal any outliers amongst the consumer liking data (XLSTAT Version 2010.2.03). In this research hierarchical methods were used as the dendrogram produced from this type of analysis provides a clearer picture of where the clusters stem from which is useful in further analysis (see Chapter 1.4.3.2). The Euclidean distance index of dissimilarity calculates the distance between two points (consumer liking scores) as the length of the hypotenuse of a right angled triangle (Pythagoras's theorem) (MacFie, 2007). This is the most commonly used raw index and was also chosen for this research. The unweighted pair-group average agglomeration method (see Chapter 1.4.3.2) was selected for this analysis as this method of identifying clusters tends to generate clusters with small within cluster variation (Hair et al., 2006). Therefore it is a more discriminating agglomeration method, useful for identifying any outliers.

In this case six consumers were identified as outliers, rating differently to the general trends (i.e. they were in individual clusters of their own); liking scores for these consumers were therefore removed and further AHC performed on the remaining data. This time the Wards agglomeration method (with the Euclidean distance index of dissimilarity) was used as it produces more compact clusters that tend to have similar numbers of consumers in each (MacFie, 2007). This is desirable for further analysis where comparison between cluster groups is more meaningful if there are similar numbers of consumers in each group (see also Chapter 1.4.3.2). Clusters were compared to determine which characteristics of the creams were segregating the consumers into groups.

Following AHC, external preference mapping was performed to determine the relationship between sensory attributes and liking and to observe the patterns of liking behaviour for different consumer groups (see also Chapter 1.4.3.3). Consumers showing different model types (vector, ideal point, saddle and anti-ideal) were compared to identify what was driving the different types of consumer liking behaviour.

Kano modelling as described in Chapter 1.4.3.4 involves separating consumer liking data into satisfaction and dissatisfaction scores. This procedure was followed for consumer study data as an alternative way of looking at the overall consumer liking. Satisfaction and dissatisfaction scores were calculated as follows; initially a threshold value was established for each individual consumer:

$$\text{Threshold} = \frac{(\text{maximum liking score} + \text{minimum liking score})}{2} \qquad (2.1).$$

The average threshold value for all consumers and average liking scores for each cream were then used to calculate average dissatisfaction (DIS) and satisfaction (SAT) scores. All creams with liking scores above the threshold were given zero for DIS and positive values for SAT, creams scoring below the threshold were given zero for SAT and positive values for DIS. For example, if the average threshold value for a consumer was 29 and their liking score for a cream was 63, then the cream would score zero for DIS and (63-29) = 34 for SAT. On the other hand if the liking score was 18, then the cream would score zero for SAT and (29-18) = 11 for DIS.

Satisfaction and dissatisfaction scores were used to plot graphs of average satisfaction/dissatisfaction data (y-axis) against sensory attributes (x-axis) so key drivers of consumer acceptability could be judged. To aid visualisation of results dissatisfaction scores were plotted as negative values. An example of a typical plot is given in Figure 2.10 which shows that consumers were generally dissatisfied with extreme samples regarding the attribute firmness i.e. samples with firmness > 8 or < 2.

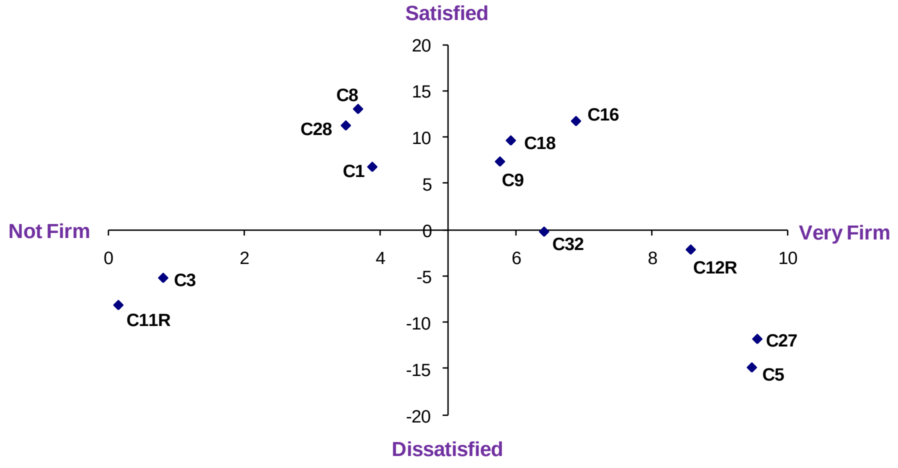


Figure 2.10: *Average dissatisfaction and satisfaction scores plotted against QDA firmness data.*

2.4 PHYSICAL METHODS

2.4.1 Rheology

The rheological behaviour of the skin creams was quantified using a rotational shear rheometer (MCR 301, Anton Paar, UK). Commercial skin creams were used to develop steady shear and oscillatory shear protocols suitable for all model skin creams despite their vast range of textures. The benefit was that the rheological parameters extracted from these measurements could be directly compared between the creams and subjected to statistical analyses that would create a link to the sensory results. There were exceptions in terms of applicability of the measurement protocol or in terms of extracting certain rheological parameters as discussed in Chapter 3.3. Measurements were carried out at 20 ºC as this corresponded to the average temperature creams were presented to panellists and consumers in the sensory studies.

The parallel plate geometry, 25 mm in diameter, was chosen for use in all tests following measurements on the commercial skin creams using a range of geometries such as different diameter parallel plates, a vane geometry and concentric cylinder geometries with smooth or rough/serrated surfaces. Serrated plates (25 mm diameter, at a gap height of 1 mm), were selected for the majority of the measurements in order to minimise slip. Thin film measurements involving smooth plates with a nominal gap height of 50 μm were applied to obtain viscosity readings at high shear rates. The small gap height meant that serrated plates were not practical in this case and slip effects are typically not observed at high shear rates.

The specifics of each test and the corresponding data analysis are described in more detail below. In all cases, samples filled into the measurement gap were allowed to rest for five minutes prior to starting the test in order to allow for temperature equilibration. The rheometer's peltier hood was used to prevent dehydration of the sample at the rim of the geometry.

2.4.1.1 Oscillatory tests

Amplitude and frequency sweeps were carried out to obtain rheological parameters which in the experience of the industrial sponsor relate to the initial application procedures, the sampling of the skin cream from its container and the overall skin cream texture (Hopkinson and Williams, 2007). The amplitude sweep also served the purpose of identifying the linear viscoelastic domain (LVD), which is important for the choice of measurement parameters in the frequency sweep. They ought to be conducted within the LVD in order to obtain results only affected by the change of frequency and not simultaneously by change of deformation or stress amplitude (Mezger, 2006).

2.4.1.1.1 Amplitude sweep

The amplitude sweeps were conducted at a frequency of 1 rad.s^{-1} while increasing the strain amplitude stepwise (8 data points/decade) from 0.1 – 1000 %. The high upper limit for the strain was deliberately chosen to ensure sufficient data points were recorded so yield stress analysis could be carried out for the range of sample properties (very thin to very thick). The test duration was 198 s, allowing 6 s per measuring point. Figure 2.11 illustrates typical results obtained for creams 11, 4 and 12R ranging from thin (C11) to thick (C12R). Data reproducibility was within 20 % for the majority of creams (34 creams) when in the LVD, while at 100 % strain, 39 samples showed < 20 % error between replicates. The limiting value of the LVD was recognised as the point where the deviation between storage modulus (G′) data points acquired at increasing deformation was greater than 10 %.

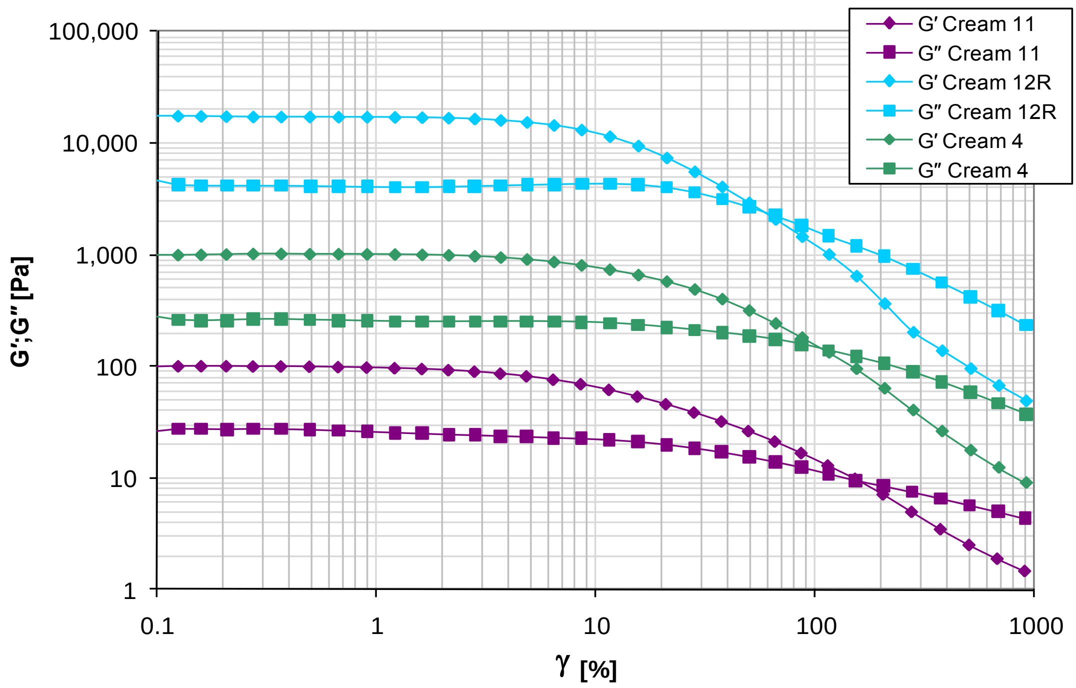


Figure 2.11: *Oscillation Amplitude Sweep results for three cream samples with differing textural properties, from thin to thick, creams 11, 4 and 12R respectively.*

Amplitude data were analysed for yield stress following the method described by Walls et al. (2003). In brief, this method consists of plotting the elastic stress (G′ multiplied by absolute strain) against the absolute strain. The maximum value in the elastic stress curve is then interpreted as the yield stress (Yang et al., 1986; Walls et al., 2003), see Figure 2.12 for an example. The yield strain values (strain at which the elastic stress is at its maximum) were also recorded for each sample to give a measure of the stretchiness of the sample (Hopkinson and Williams, 2007), see Figure 2.12. Averaged experimental results were used to plot these curves. Further parameters extracted from the amplitude sweep data were the storage modulus (G′), the loss modulus (G″) and the complex viscosity (η*) at 0.1 %, 1 % and 100 % strain. The values at different strains were extracted so that cream behaviour under different levels of shear deformation could be compared.

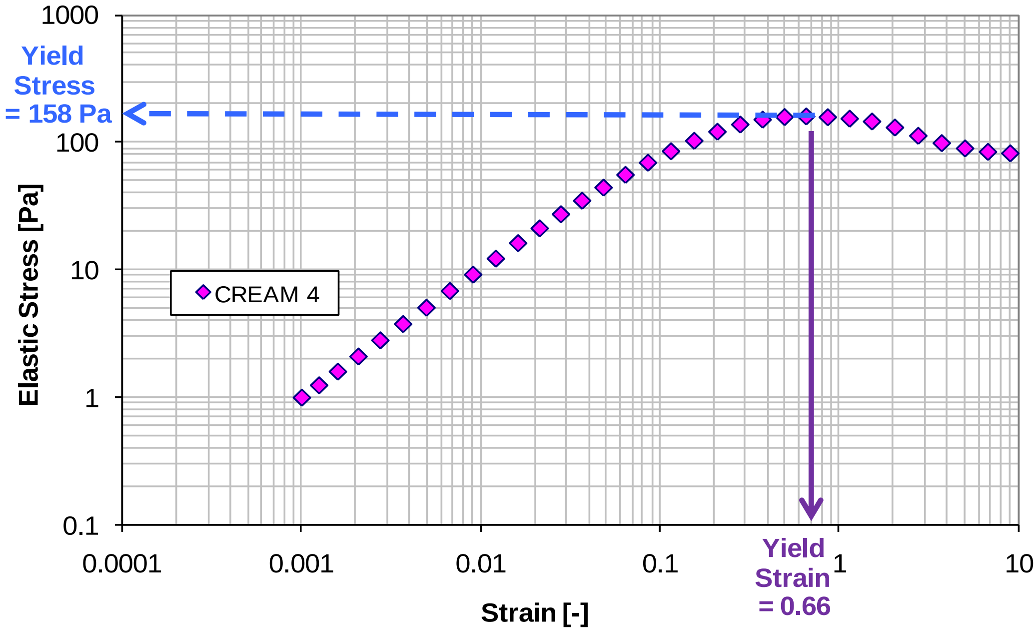


Figure 2.12: *Analysis of amplitude sweep data for yield stress and yield strain, where elastic stress = G′ multiplied by absolute strain. Note in this Figure the strain plotted represents the absolute strain hence the units [-].*

2.4.1.1.2 Frequency sweep

Frequency sweeps were carried out at 1 % strain, which was within the LVD for all creams, and the angular frequency was increased from 0.1 - 10 rad.s^{-1} recording 8 data points/decade. The measurement point duration was set to decrease logarithmically from 60 to 6 s. Triplicate data for each sample was averaged and then analysed as follows: logG′-logω and logG″-logω plots were generated and the data were fitted with a linear equation using Microsoft Office Excel 2003. The slopes and intercepts of these lines were recorded, see example in Figure 2.13 in which the slope for G′ is 0.146 and the intercept is 3.7 whereas the slope for G″ is 0.086 and the intercept is 3.3. Additionally the values for G′, G″ and tanδ (tanδ = G″ / G′) at 1 rad.s^{-1} were all calculated based on averaged results. It should be noted that for measurements on the subset of model skin creams used in the consumer study, frequency was varied between 0.1 -100 rad.s^{-1} to cover a wider frequency range thus potentially improving detection of differences between individual samples.

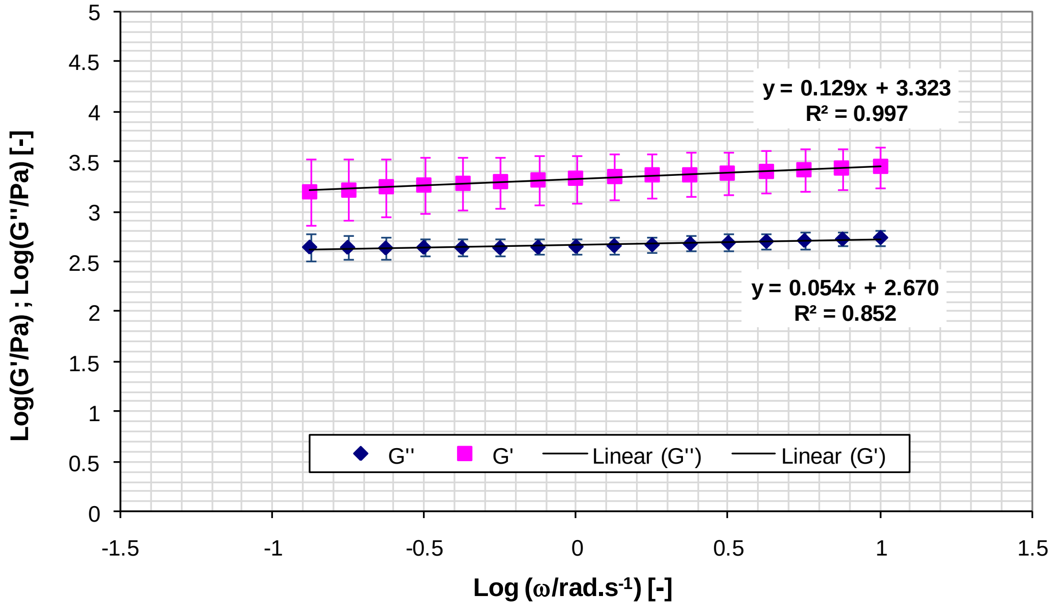


Figure 2.13: *Example of linear regression procedure for calculating the slope of [log G′ vs. log ω] and [log G″ vs. log ω] curves from frequency sweep data. Standard deviations are given in the form of error bars.*

2.4.1.2 Steady shear tests

Steady shear measurements were conducted to quantify the shear viscosity of the cream samples. The flow behaviour of the creams was highly shear thinning over a narrow shear stress range thus required application of separate test protocols to assess viscosity at low and high shear respectively.

2.4.1.2.1 Low shear viscosity data acquisition and analysis

Low shear viscosity was quantified by applying a continuous shear stress ramp between 1 and 10^4 Pa acquiring 10 logarithmically spaced points per decade. Total test duration was 164 s within which 31 data points were recorded. For this test 25 mm serrated parallel plates were used with a gap height of 1 mm. Data was analysed for apparent yield stress using the rheometer supplier's software (Rheoplus/32 Version 3.21, 2007) which uses regression to calculate the bending point of a stress – viscosity curve in a logarithmic plot. The data point with the largest distance to the regression lines is taken as the yield stress (Anton-Paar, 2007).

Plotting the viscosity results as a function of shear rate revealed a viscosity curve with a zero shear viscosity plateau (η_0). To extract a value for η_0, the curves were fitted with the Cross model (Cross, 1965), see Equation (2.2), using the rheometer supplier's software. Noisy data at very low shear rates was excluded from the analysis. The viscosity curves did not show a plateau value at high shear rates which is a parameter in the Cross model (η_∞). However, since the data showed an inflexion point in the shear-thinning region, unsatisfactory regression coefficients were obtained with a rheological model considering only a low shear viscosity plateau such as the Ellis model (Mezger, 2006). The Cross model parameters were calculated for each replicate of each sample and results averaged for comparison between creams (Chapter 3.3.3). An example of the Cross model fitting data is given in Figure 2.14 while the equation follows:

$$\eta = \frac{\eta_0 - \eta_\infty}{1 + (\mathrm{a} \cdot \dot{\gamma})^{\mathrm{p}}} + \eta_\infty \tag{2.2}$$

where η_0 is the zero shear viscosity (Pa.s); η_∞ the infinite shear viscosity (Pa.s); $\dot{\gamma}$ the shear rate (s^{-1}); a the Cross time constant (s); and p the Cross rate constant (dimensionless).

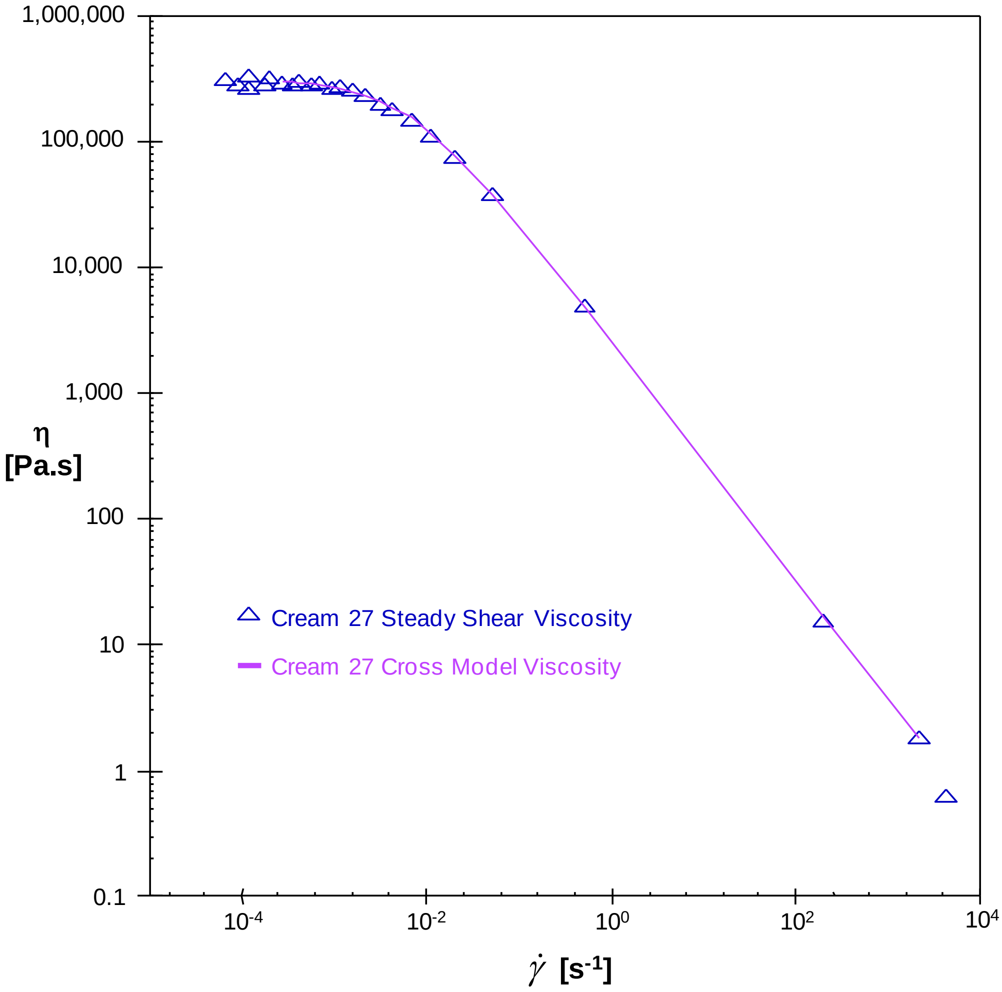


Figure 2.14: *Example of the Cross model (line) fitting a set of data from the steady shear measurements (open, triangle data points).*

2.4.1.2.2 High shear viscosity data acquisition and analysis

Viscosity data at high shear were acquired since in the later stages of skin cream application high shear rates are employed (10 - 10^4 s^{-1}) as the sample thickness decreases (20 - 100 μm) (Pham, 2000). To apply high shear rates the technique of thin film rheology using smooth parallel plates with a nominal gap height of 50 μm was used. The shear rate was increased stepwise from nominally 0.01 - 10^6 s^{-1} acquiring 10 logarithmically spaced points per decade. At such narrow gaps acquired viscosity data needs to be corrected for gap height and the non-Newtonian nature of the creams demands a further correction (Kramer et al., 1987;

Shaw and Liu, 2006; Davies and Stokes, 2008). For both corrections previously developed protocols were applied, these are outlined in Appendix VII.

The majority of thin film rheology results showed that the upper limit was not reached due to the thinning properties of the creams which lead to spinning through of the rotating plate and any further data collected was not meaningful. Repeat measurements were therefore not taken for the majority of the skin creams (this test only). A comparison between samples was made using the viscosity at 10,000 s^{-1} (from the up curve) and the highest useful shear rate recorded per cream (these results were taken directly from the data tables generated during the measurement by the computer software).

The thin film measurement protocol described above was modified slightly to measure the subset of model skin creams used in the consumer study. The shear rate was increased from 1000 - 1,000,000 s^{-1}, collecting 4 data points per decade – this decreased the measurement time which allowed more reproducible data to be obtained.

2.4.1.3 Statistical analysis of rheological data

For the test methods described above triplicate data was averaged and standard deviations calculated. Using this averaged data, two types of analysis were carried out. Principal component analysis (PCA) was used to visualise the relationship between the rheological parameters and sensory attributes (XLSTAT, Version 2007.6) and polynomial predictive modelling (Design Expert software, version 6.0.6, 2000) was used to determine if rheological models could be constructed to predict sensory (textural) properties of skin cream (see Chapter 4).

2.4.2 Texture analysis

The TA.XT plus Texture Analyser (Stable Micro Systems) was used to characterise the subset of 12 model skin cream samples used in the consumer study. Based on preliminary tests the back extrusion method as defined by the instrument's manual was selected as the most suitable for obtaining reproducible data relevant to skin cream usage. It also supplies data that the other physical measurements in this research could not provide such as a measure of the 'cohesiveness' of the sample. Other parameters measured by this test include the firmness, consistency and the index of viscosity (definitions follow).

For each cream sample, six replicates were taken. Samples were measured in 100 mL polypropylene containers (height 72 mm, top diameter 56 mm, base diameter 45 mm) holding 60 mL cream (~33 mm 'fill height'). A cylindrical aluminium probe (38 mm diameter, 5 mm height) was fitted to the measurement arm of the texture analyser. The probe was inserted 25 mm into the sample at a speed of 2 $mm.s^{-1}$. On lowering the probe into the sample, the forces measured were used to calculate the **firmness** and **consistency** of the samples, on withdrawal of the probe from the sample (speed 2 $mm.s^{-1}$) the **index of viscosity** and **cohesiveness** were recorded. Texture Exponent 32 software (version 2,0,6,0) was used to calculate the values for these parameters, see Figure 2.15 for illustration.

Firmness is defined as the maximum positive force recorded when the probe reaches the chosen maximum depth (i.e. 25 mm). The higher the force value, the firmer the sample. The consistency relates to the area under the curve up to the maximum force reading, the higher the value, the thicker the sample. The cohesiveness is evaluated as the minimum force reading, the more negative this value, the more cohesive the sample. Finally the area of the negative region of the curve is interpreted as the index of viscosity (Stable-Micro-Systems, 2005), see Figure 2.15. The units are g and g.s for the force parameters (firmness and cohesiveness) and the area parameters (cohesiveness and index of viscosity) respectively.

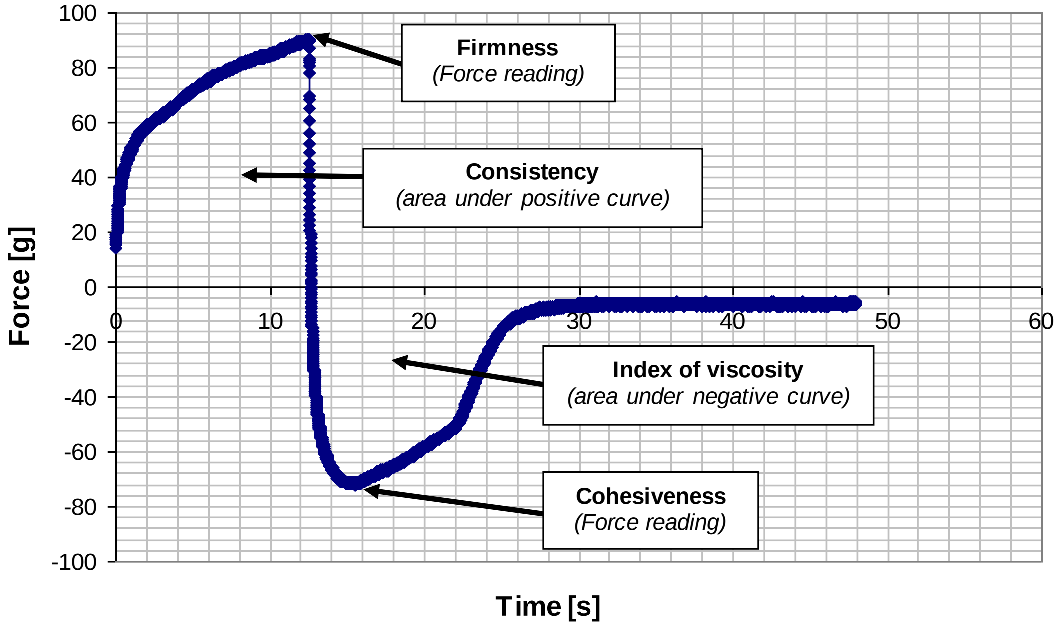


Figure 2.15: *Example of a back extrusion result for a skin cream. Test parameters derived from the curve are also shown.*

2.4.2.1 Statistical analysis of back extrusion data

Firmness, consistency, index of viscosity and cohesiveness results for the 6 replicates were averaged and standard deviations calculated. The relationship between sensory attributes and parameters from texture analysis was determined using principal component analysis *(XLSTAT, Version 2007.6).*

2.4.3 Force plate measurements

Force plate analysis was performed on the subset of 12 model skin cream samples used in the consumer study. This relatively new technique developed by Andrew Hopkinson and Adrian Williams (Unilever Plc) involved a panellist spreading cream samples (using their index finger) on a plate resting on a platform that recorded the forces exerted (Hopkinson et al., 2008). The force, **F** = (Fx,Fy,Fz), was measured in three directions: two horizontal axes (Fx and Fy) and one vertical axis (Fz) which represents the load, see Figure 2.16. The torque, **T** = (Tx,Ty,Tz), was also measured (Tx – torque around the x-axis, Ty – torque around the y-axis and Tz

– torque around the z axis). For details on how these variables were measured see Hopkinson et al. (2008).

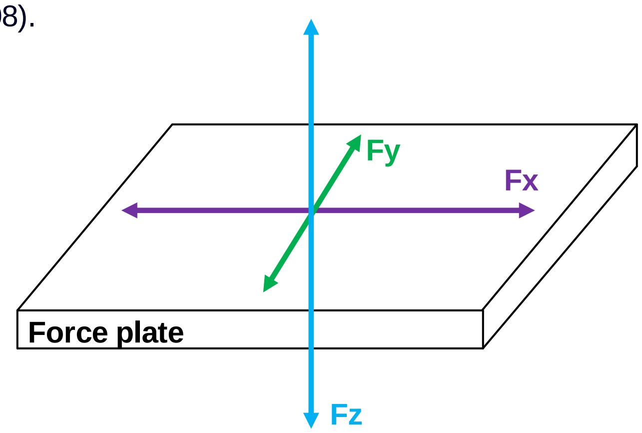


Figure 2.16: *Diagram illustrating the forces measured during force plate analysis where Fz = the force in the vertical axis (the load) and Fy and Fx, the forces in the horizontal axes.*

Using normal load (Fz) and torque data from the horizontal axes (Tx & Ty) the position of the panellists finger (x, y) from the centre of the plate could be calculated (Hopkinson et al., 2008), since

$$x = \frac{Ty}{Fz} \qquad (2.3) \qquad\qquad y = \frac{Tx}{Fz} \qquad (2.4).$$

The speed at which the finger was moving could then be found from the derivative of the position with time. Examples of the typical forces and torques involved in stroking measurements are given in Figures 2.17 and 2.18. Finger position calculations (as described above) were made using this type of data.

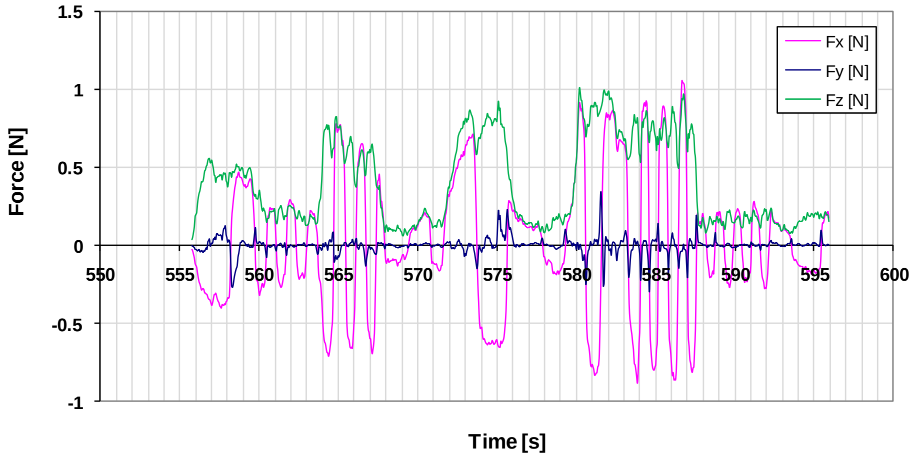


Figure 2.17: *Forces involved in a typical stroking measurement (results obtained for cream 27, episode 6 are shown).*

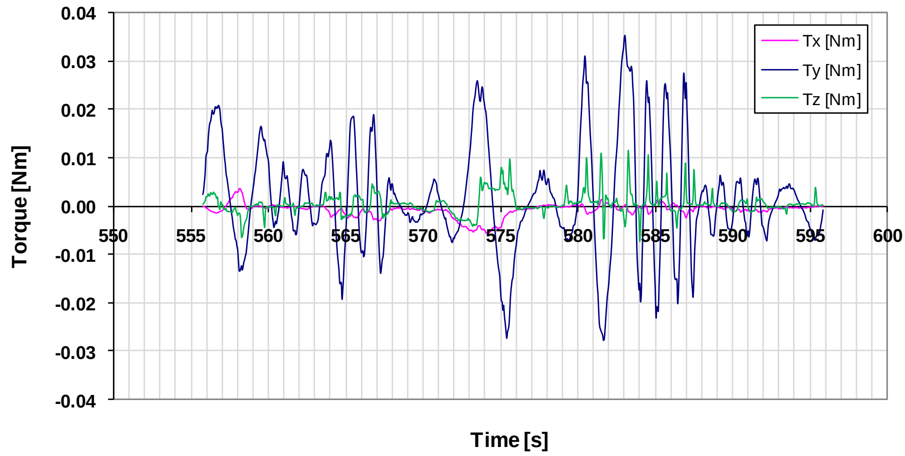


Figure 2.18: *Torques obtained during a stroking measurement for cream 27, episode 6.*

In this study, one 25-year-old female panellist characterised the frictional properties of all the consumer study creams. Bioskin (synthetic skin, polyurethane, Japan, ex S.Black, UK) was mounted onto the force plate to allow for more realistic friction readings to be gained. For each measurement 30.5 μL[1] of cream was pipetted onto the Bioskin (area 12 x 2 cm^2) and spread out by the panellist to cover the Bioskin evenly prior to data recording. Stroking measurements were then carried

[1] Note that this volume is greater than that used by the trained panel when rating attributes on PC2, but the aim was to use a volume that would result in a similar film thickness being applied to the Bioskin. The trained panel used a smaller volume of cream spread over a smaller area which equated to a similar film thickness to that used in this experiment.

out; each measurement lasted 10 minutes during which 6 episodes of data were recorded. Each stroking episode lasted 40 seconds and the intervals between episodes were not evenly spaced to account for more changes in the early stages of stroking compared to the later stages (see Table 2.4). This allowed the effects of drying of the cream sample on the apparent friction between finger and skin to be analysed.

Table 2.4: *Start times for stroke episodes illustrating the difference in intervals between episodes.*

<table>
<tr><th>Episode</th><th>Start time of episode [s]</th><th colspan="5">Interval between episodes [s]</th></tr>
<tr><td>1</td><td>30</td><td rowspan="2">20</td><td></td><td></td><td></td><td></td></tr>
<tr><td>2</td><td>90</td><td rowspan="2">20</td><td></td><td></td><td></td></tr>
<tr><td>3</td><td>150</td><td></td><td rowspan="2">50</td><td></td><td></td></tr>
<tr><td>4</td><td>240</td><td></td><td></td><td rowspan="2">90</td><td></td></tr>
<tr><td>5</td><td>370</td><td></td><td></td><td></td><td rowspan="2">145</td></tr>
<tr><td>6</td><td>555</td><td></td><td></td><td></td><td></td></tr>
</table>

The stroking test involved the panellist spreading the sample back and forth on the Bioskin (using the index finger) following a prompt on the computer screen indicating a target of how fast to stroke and how much pressure to apply. The actual load and speed were also plotted so they could be adjusted to be closer to the target. An example of the display the panellist could see is given in Figure 2.19. The target (large circle) moved around the screen indicating which load-speed ranges required more data points. The example shows that the panellist is asked to apply a heavy load and to stroke back and forth slowly. It also shows that the actual load and speed being applied by the panellist (small circles) at that point in time was heavy and fast respectively.

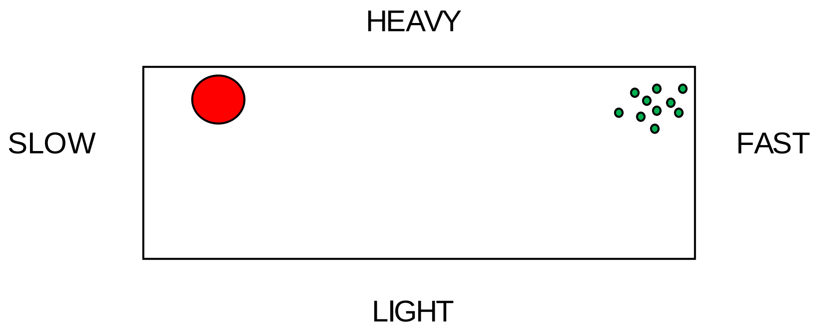


Figure 2.19: *Example of prompt viewed by the panellist to influence speed and pressure used when stroking during force plate measurements. The large red circle indicates the target load and speed to be applied, while the small green circles indicate the actual load and speeds being applied.*

Speeds in the range 10 - 500 $mm.s^{-1}$ and normal loads in the range 0.02 - 5 N were recorded. All measurements were carried out in an indirectly controlled room (a room adjacent to a lab with comfort air conditioning which provided a degree of temperature control), relative humidity and temperature readings were recorded following each measurement. Duplicate measurements were carried out for each cream (time limitations, in terms of instrument availability, meant that further replicates could not be made).

2.4.3.1 Theoretical background to the force plate measurements

Friction forces involved in skin cream application are very important with regard to the overall sensory perception of the cream (Gee et al., 2005). Generally, when moving an object across a surface, the force resisting motion is called friction. In this experiment the friction force is defined as the magnitude of the force in the x-y plane:

$$Friction = \sqrt{Fx^2 + Fy^2} \qquad (2.5).$$

It is clear that the properties of the contacting materials, and the normal force applied (load), will change the friction force, therefore it is usual to also define:

$$Friction = \mu \times Load = \mu Fz \qquad (2.6)$$

where μ is the friction coefficient (see also Chapter 1.7.1). Note that for slippery creams, a low friction coefficient value and therefore low friction would be expected, whereas if there was no cream between the surfaces, the opposite would apply. In order to understand these relationships the following graphs can be plotted: friction against load or speed, friction coefficient against load or speed and friction coefficient against speed/load (Stribeck curve), see Chapters 1.7 and 3.5.

2.4.3.2 Force plate data analysis

Once data had been collected, results were divided up into 50 ms snippets and analysed. Raw data was checked for consistency amongst replicates and any other sources of error. Then reduction methods were used to convert the large raw data set into more manageable sized chunks for comparisons and further analysis. Speed and load ranges of interest were 10 - 500 $mm.s^{-1}$ and 0.02 - 5 N respectively; therefore any data recorded outside of these ranges were discarded. These ranges cover the normal pressures that people exert when making touch-feel assessments (Liu et al., 2008) and the speeds occurring during skin cream application (Brummer and Godersky, 1999). Friction coefficient and friction for the different creams were calculated and visualised with Spotfire (visualisation software). An example of the range of loads and speeds obtained for a typical cream during one episode is given in Figure 2.20. Each data point represents 50 ms of data.

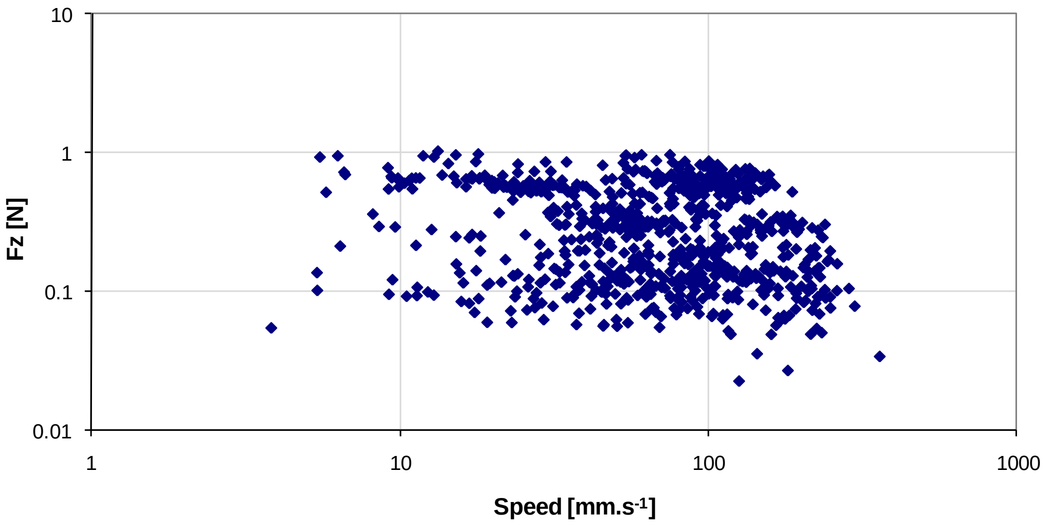


Figure 2.20: *Range of loads and speeds obtained during episode 6 for a single replicate of cream 27.*

After initial observations checking consistency between replicate data, further data reduction was carried out resulting in a data set containing nine combinations of load and speed ranges (see Table 2.5).

Table 2.5: *Load-speed ranges from which the main force plate data analysis was conducted.*

Load-speed range	Load [N]	Speed [mm.s^{-1}]
1	0.1 – 0.2	20 – 50
2	0.2 – 0.5	20 – 50
3	0.5 – 1	20 – 50
4	0.1 – 0.2	50 – 100
5	0.2 – 0.5	50 – 100
6	0.5 – 1	50 – 100
7	0.1 – 0.2	100 – 200
8	0.2 – 0.5	100 – 200
9	0.5 – 1	100 – 200

Medians, means and quartiles of selected log scaled variables were calculated from these nine load-speed ranges. These medians were used to calculate the Stribeck slope, the log(Fz) factor, the log(Speed) factor and the log(Coefficient) factor (at 100 mm.s^{-1} and 0.5 N) for each episode. The **Stribeck slope** was found from the linear fit of the log(friction coefficient) versus log(speed/load) plot using the medians obtained from the nine load-speed ranges. The **log(Fz) factor** and the **log(Speed) factor** were slope values as obtained through linear fits of the planes log(friction coefficient) versus log(Fz) and log(friction coefficient) versus log(speed) respectively. Least squares minimisation was used to fit these planes. The **log(Coefficient) factor** combines both Speed and Fz factors giving an estimate of the overall frictional properties of the creams at 100 $mm.s^{-1}$ and 0.5 N. It was calculated using the following equation:

log(Coefficient) factor at 100 $mm.s^{-1}$ and 0.5 N

$$= \text{constant} + \mathbf{Fz(factor)} * \log(0.5) + \mathbf{Speed(factor)} * \log(100) \qquad (2.7).$$

This coefficient at a typical load and speed (100 $mm.s^{-1}$ and 0.5 N) as well as the Fz and Speed factors were useful for determining certain characteristics of the skin creams. For example, the Stribeck slope gave an idea of the film thickness between the two surfaces by indicating which lubrication regime the sample was in (see Chapter 1.7.1). The Fz, Speed and Coefficient factors allowed judgements about the overall slipperiness of the samples to be made (see Chapters 1.7 and 2.4.3.1). Results were correlated to the trained panel sensory data through PCA and predictive modelling (see Chapter 4).

3. RESULTS AND DISCUSSION

3.1 SENSORY RESULTS

3.1.1 Textural properties of 40 model skin creams

The textural properties of the 40 model skin creams were rated by the trained panel for the following attributes: firmness, thickness, resistance, spreadability, stickiness, cooling, drying, dragging, slipperiness, final greasiness and absorption according to the measurement protocols given in Table 2.3, Chapter 2.3.2.3. Results were analysed as described in Chapter 2.3.2.8.

Two-way ANOVA showed significant panellist, product and panellist-product interactions ($p < 0.05$). This can be explained by the large number of samples assessed for each attribute therefore where significant differences occur between many samples, similarities also occur between others for the assessed attributes. This can lead to cross-over interactions between panellists which are common in sensory science even with trained panels. A high level of cross-over may be observed when the panellists are not following a similar rating pattern for example in Appendix 3, Figure A3.1, it is clear that the panellist represented with dark blue diamonds is rating the samples differently to the other panellists, hence the large degree of cross-over observed. On the other hand in Figure A3.2, all panellists are showing a similar pattern of liking behaviour therefore the level of cross-over is limited. However, cross-over is still observed which is why in some cases significant panellist-product interactions are recorded. In this research, due to the large number of samples, and the similar pattern of liking behaviour observed in the interaction plots, these cross-over interactions were considered to be at an acceptable level.

Tukey's HSD post hoc test revealed that panellists were able to differentiate across the 40 skin creams for the different attributes. The largest number of homogeneous subsets panellists separated samples into was 21 (out of a maximum of 40). Results are summarised in Tables 3.1 - 3.4 where for each attribute mean panel scores, Tukey's homogeneous subsets and standard deviations are given. Tukey's homogeneous subsets results illustrate significant differences between creams for the different attributes. These differences are indicated by letters, where for individual attributes samples with the same letter were not significantly different to each other ($p > 0.05$). For example looking at firmness creams 1 and 6 were not significantly different to each other (both have G and H in common), whereas creams 1 and 2 were significantly different to each other in firmness (see Table 3.1).

Table 3.1: *Mean scores, standard deviations and homogeneous subsets obtained through rating of the 40-model skin creams by the trained panel for the attributes firmness, thickness, resistance and spreadability. Samples with the same letter, within a column are not significantly different to each other (p > 0.05).*

Cream	FIRMNESS		THICKNESS		RESISTANCE		SPREADABILITY	
	Mean & Tukey's homogen - eous subsets	***SD***	**Mean & Tukey's homogen-eous subsets**	***SD***	**Mean & Tukey's homogen - eous subsets**	***SD***	**Mean & Tukey's homogen-eous subsets**	***SD***
C1	4.2 ^{GHIJ}	*2.7*	4.7 ^{GHI}	*2.6*	3.3 ^{GH}	*3.1*	3.2 ^{EFG}	*2.8*
C2	0.2 ^{A}	*0.3*	0.4 ^{A}	*0.5*	0.2 ^{A}	*0.3*	0.1 ^{A}	*0.2*
C3	1.9 ^{CDE}	*1.6*	2.4 ^{DE}	*1.7*	0.9 ^{ABCD}	*1.3*	0.6 ^{ABC}	*1.0*
C3R	1.7 ^{BCD}	*1.3*	2.5 ^{DE}	*1.7*	0.8 ^{ABCD}	*1.0*	0.4 ^{AB}	*0.6*
C4	6.0 ^{LM}	*2.1*	6.3 ^{KL}	*2.1*	5.1 ^{IJKL}	*2.2*	4.2 ^{FGHIJ}	*2.1*
C4R	5.8 ^{KLM}	*1.7*	6.0 ^{JKL}	*1.6*	4.9 ^{IJKL}	*1.8*	3.8 ^{FGHI}	*1.8*
C5	9.3 ^{T}	*1.0*	9.4 ^{U}	*1.0*	9.4 ^{T}	*0.6*	8.9 ^{P}	*1.1*
C5R	8.9 ^{RST}	*1.3*	9.1 ^{STU}	*1.2*	8.4 ST	*1.5*	8.2 ^{OP}	*1.7*
C6	3.3 ^{FGH}	*2.1*	3.9 ^{FG}	*1.9*	1.5 ^{BCDEF}	*1.5*	0.9 ^{ABC}	*1.1*
C7	8.3 ^{PQRST}	*1.4*	8.4 ^{PQRSTU}	*1.4*	7.1 ^{OPQR}	*1.7*	5.9 ^{LM}	*2.5*
C7R	8.3 ^{PQRST}	*1.5*	8.3 ^{PQRSTU}	*1.5*	6.7 ^{NOPQR}	*1.8*	5.1 ^{IJKLM}	*2.2*
C8	3.1 ^{EFG}	*2.5*	3.8 ^{FG}	*2.5*	1.2 ^{ABCDE}	*1.4*	0.8 ^{ABC}	*0.8*
C8R	2.9 ^{DEF}	*2.6*	3.7 ^{FG}	*2.5*	1.3 ^{ABCDE}	*1.7*	0.7 ^{ABC}	*0.9*
C9	5.7 ^{KLM}	*2.8*	6.2 ^{JKL}	*2.8*	4.9 ^{IJKL}	*2.4*	4.7 ^{HIJKL}	*2.3*
C10	1.1 ^{ABC}	*0.9*	1.6 ^{BCD}	*1.3*	0.6 ^{ABCD}	*0.9*	0.4 ^{AB}	*0.7*
C11	1.3 ^{ABC}	*1.1*	1.8 ^{CD}	*1.3*	0.4 ^{ABC}	*0.4*	0.3 ^{AB}	*0.3*
C11R	0.4 ^{A}	*0.4*	0.6 ^{AB}	*0.6*	0.3 ^{AB}	*0.5*	0.2 ^{A}	*0.3*
C12	8.3 ^{PQRST}	*1.4*	8.3 ^{PQRSTU}	*1.4*	6.5 ^{NOPQ}	*2.0*	5.1 ^{IJKLM}	*2.7*
C12R	8.5 ^{QRST}	*1.2*	8.3 ^{PQRSTU}	*1.2*	6.0 ^{LMNO}	*2.1*	4.4 ^{GHIJK}	*2.3*
C14	8.1 ^{PQRS}	*1.9*	8.2 ^{PQRST}	*1.8*	4.6 ^{IJK}	*2.8*	3.5 ^{FGH}	*2.6*
C15	8.5 ^{QRST}	*1.8*	8.9 ^{RSTU}	*1.1*	7.8 ^{QRS}	*1.9*	6.4 ^{MN}	*2.3*
C16	6.6 ^{MNO}	*1.7*	7.0 ^{LMNO}	*1.7*	5.6 ^{JKLMN}	*1.8*	3.8 ^{FGHI}	*2.0*
C17	8.8 ^{RST}	*1.2*	8.7 ^{QRSTU}	*1.1*	6.5 ^{MNOP}	*2.2*	5.3 ^{JKLM}	*2.2*
C18	7.4 ^{OPQ}	*1.8*	7.6 ^{NOPQ}	*1.8*	5.2 ^{JKLM}	*2.3*	3.8 ^{FGHI}	*2.1*
C20	6.6 ^{MNO}	*2.1*	6.8 ^{LMNO}	*2.1*	3.9 ^{HI}	*2.4*	2.8 ^{DEF}	*1.8*
C23	6.2 ^{LMN}	*2.4*	6.6 ^{KLM}	*2.2*	4.5 ^{HIJ}	*3.2*	5.8 ^{KLM}	*2.8*
C24	7.7 ^{OPQR}	*1.6*	7.9 ^{OPQRS}	*1.7*	6.7 ^{NOPQR}	*1.7*	5.6 ^{KLM}	*2.3*
C25	4.2 ^{GHIJ}	*2.2*	4.3 ^{FGHI}	*2.2*	1.6 ^{BCDEF}	*1.8*	1.0 ^{ABC}	*1.0*
C27	9.1 ST	*1.1*	9.2 ^{TU}	*1.1*	8.6 ST	*1.4*	7.7 ^{NOP}	*2.1*
C28	3.9 ^{FGHI}	*2.4*	4.3 ^{FGH}	*2.4*	1.7 ^{DEF}	*2.5*	1.3 ^{ABC}	*1.8*
C29	4.5 ^{HIJK}	*2.6*	5.1 ^{HIJ}	*2.6*	2.4 ^{EFG}	*2.6*	1.7 ^{BCD}	*1.7*
C30	7.7 ^{OPQR}	*1.8*	7.9 ^{OPQR}	*1.8*	7.4 ^{PQRS}	*1.8*	7.4 ^{NO}	*2.2*
C31	0.5 ^{AB}	*0.6*	0.9 ^{ABC}	*0.9*	0.3 ^{AB}	*0.5*	0.2 ^{A}	*0.3*
C32	5.2 ^{JKL}	*2.8*	6.0 ^{JKL}	*2.5*	4.4 ^{HIJ}	*2.4*	3.6 ^{FGH}	*2.1*
C33	7.1 ^{NOP}	*2.2*	7.5 ^{MNOP}	*2.0*	5.9 ^{KLMN}	*2.3*	4.6 ^{GHIJKL}	*2.1*
C34	5.1 ^{IJKL}	*2.6*	5.5 ^{IJK}	*2.5*	2.6 ^{FG}	*3.0*	2.8 ^{DEF}	*2.5*
C35	6.3 ^{LMN}	*2.0*	6.4 ^{KLM}	*2.0*	2.2 ^{EFG}	*2.3*	1.9 ^{CDE}	*2.2*
C36	3.3 ^{FGH}	*2.0*	3.8 ^{FG}	*2.1*	1.7 ^{CDEF}	*2.0*	1.2 ^{ABC}	*1.3*
C37	3.2 ^{FG}	*1.8*	3.4 ^{EF}	*1.9*	1.4 ^{ABCDEF}	*1.5*	0.9 ^{ABC}	*1.0*
C40	7.8 ^{OPQRS}	*2.8*	8.9 ^{RSTU}	*1.0*	7.8 ^{RS}	*1.8*	7.4 ^{NO}	*2.1*

Table 3.2: *Mean scores, standard deviations and homogeneous subsets obtained through rating of the 40-model skin creams by the trained panel for the attributes stickiness and cooling. Samples with the same letter, within a column are not significantly different to each other (p > 0.05).*

Cream	STICKINESS		COOLING	
	Mean & Tukey's homogeneous subsets	***SD***	**Mean & Tukey's homogeneous subsets**	***SD***
C1	4.6 BCDEFGHIJ	*3.3*	3.4 A	*3.1*
C2	2.8 ABC	*3.2*	5.9 AB	*3.5*
C3	3.6 BCDEF	*3.1*	5.3 ABC	*3.0*
C3R	4.7 CDEFGHIJ	*3.1*	5.2 PQ	*2.7*
C4	6.2 IJKL	*2.4*	3.7 ABCD	*2.8*
C4R	5.6 FGHIJKL	*3.0*	3.1 OPQ	*3.1*
C5	5.8 GHIJKL	*3.4*	1.7 ABCDE	*2.3*
C5R	5.3 EFGHIJK	*3.1*	1.8 CDEFGHIJKLMNO	*2.4*
C6	3.0 ABC	*2.9*	3.9 ABCDEF	*3.1*
C7	6.9 KL	*2.1*	3.7 ABCDEF	*2.9*
C7R	6.4 JKL	*2.7*	3.2 CDEFGHIJKLMNO	*2.7*
C8	2.9 ABC	*2.9*	4.0 ABCDEFG	*3.1*
C8R	3.1 ABC	*2.8*	3.9 DEFGHIJKLMNO	*3.3*
C9	4.4 BCDEFGHIJ	*2.9*	2.2 ABCDEFG	*2.8*
C10	3.8 BCDEFGH	*3.5*	5.3 ABCDEFGH	*3.4*
C11	2.9 ABC	*2.9*	4.9 ABCDEFGHI	*3.5*
C11R	2.6 AB	*3.0*	5.4 ABCDEFGHIJK	*3.3*
C12	7.1 KL	*2.7*	4.3 ABCDEFGHIJ	*3.4*
C12R	6.9 KL	*2.1*	4.8 FGHIJKLMNOPQ	*3.1*
C14	1.4 A	*2.0*	1.2 ABCDEFGHIJKL	*1.8*
C15	5.9 HIJKL	*3.2*	2.9 ABCDEFGHIJKL	*3.0*
C16	5.8 HIJKL	*3.2*	3.1 ABCDEFGHIJKL	*2.9*
C17	5.3 DEFGHIJK	*3.0*	3.0 BCDEFGHIJKLM	*2.6*
C18	5.4 EFGHIJKL	*3.0*	3.1 CDEFGHIJKLMN	*2.5*
C20	5.7 FGHIJKL	*2.4*	4.9 CDEFGHIJKLMNO	*2.5*
C23	5.4 EFGHIJKL	*3.2*	2.1 DEFGHIJKLMNO	*2.7*
C24	5.9 HIJKL	*2.7*	2.6 EFGHIJKLMNOP	*3.3*
C25	4.0 BCDEFGH	*3.0*	5.1 FGHIJKLMNOP	*3.1*
C27	7.5 L	*2.5*	3.7 GHIJKLMNOPQ	*3.2*
C28	2.7 ABC	*3.3*	2.8 HIJKLMNOPQ	*3.1*
C29	3.7 BCDEFG	*2.8*	3.7 IJKLMNOPQ	*3.0*
C30	5.3 DEFGHIJK	*2.6*	4.1 JKLMNOPQ	*3.1*
C31	2.9 ABC	*3.1*	6.0 KLMNOPQ	*2.9*
C32	4.4 BCDEFGHIJ	*3.0*	1.9 KLMNOPQ	*2.3*
C33	5.5 FGHIJKL	*2.7*	4.7 LMNOPQ	*3.3*
C34	3.8 BCDEFGH	*3.2*	3.2 MNOPQ	*3.1*
C35	3.2 ABCD	*3.2*	2.1 NOPQ	*2.3*
C36	2.5 AB	*3.3*	2.1 OPQ	*2.7*
C37	4.2 BCDEFGHI	*3.2*	5.2 OPQ	*3.0*
C40	3.3 ABCDE	*3.1*	1.2 Q	*1.6*

Table 3.3: *Mean scores, standard deviations and homogeneous subsets obtained through rating of the 40-model skin creams by the trained panel for the attributes drying and dragging. Samples with the same letter, within a column are not significantly different to each other (p > 0.05).*

Cream	DRYING		DRAGGING	
	Mean & Tukey's homogeneous subsets	***SD***	**Mean & Tukey's homogeneous subsets**	***SD***
C1	5.0 HIJKL	*3.0*	5.3 LMNOPQR	*2.9*
C2	3.9 EFGHIJ	*3.0*	5.2 LMNOPQ	*3.3*
C3	5.4 IJKLM	*2.9*	5.5 MNOPQR	*3.1*
C3R	5.8 JKLM	*3.2*	6.4 OPQR	*2.9*
C4	2.0 ABCDE	*2.8*	2.3 ABCDEFGHI	*3.2*
C4R	1.2 ABC	*2.1*	2.2 ABCDEFGH	*2.9*
C5	2.6 BCDEFG	*2.7*	3.7 GHIJKLM	*3.1*
C5R	1.9 ABCDE	*1.8*	3.4 FGHIJKL	*2.9*
C6	2.1 ABCDEF	*2.4*	1.5 ABCDEF	*1.5*
C7	1.3 ABCD	*1.5*	2.2 ABCDEFGH	*2.1*
C7R	2.3 ABCDEF	*2.4*	2.7 DEFGHIJ	*2.6*
C8	3.5 DEFGHI	*3.0*	2.4 ABCDEFGHI	*2.5*
C8R	3.9 EFGHIJ	*3.2*	2.7 CDEFGHIJ	*2.9*
C9	1.4 ABCD	*1.6*	1.5 ABCDEF	*1.3*
C10	4.3 FGHIJ	*3.6*	4.4 IJKLMNO	*3.6*
C11	2.5 ABCDEF	*3.0*	2.5 BCDEFGHIJ	*3.0*
C11R	2.4 ABCDEF	*2.5*	1.9 ABCDEFGH	*2.1*
C12	6.6 KLM	*3.1*	7.0 QR	*2.5*
C12R	6.7 KLM	*3.1*	6.6 PQR	*2.9*
C14	0.5 A	*0.9*	0.6 ABC	*1.0*
C15	1.2 ABC	*1.5*	1.9 ABCDEFG	*2.0*
C16	0.9 ABC	*1.2*	1.1 ABCDE	*1.4*
C17	1.5 ABCD	*2.0*	2.0 ABCDEFGH	*2.1*
C18	2.7 BCDEFG	*3.2*	3.5 FGHIJKLM	*3.3*
C20	7.2 M	*2.6*	7.1 QR	*2.6*
C23	2.5 ABCDEF	*2.3*	3.3 FGHIJKL	*2.9*
C24	1.8 ABCDE	*2.2*	2.5 BCDEFGHI	*2.6*
C25	1.4 ABCD	*2.4*	1.5 ABCDEF	*2.3*
C27	7.0 LM	*2.7*	7.4 R	*2.4*
C28	0.4 A	*0.6*	0.3 A	*0.6*
C29	3.8 EFGHIJ	*3.2*	4.6 JKLMNOP	*2.9*
C30	5.1 HIJKLM	*3.1*	4.0 HIJKLMN	*2.9*
C31	4.7 GHIJK	*3.1*	5.7 NOPQR	*3.6*
C32	0.6 AB	*1.0*	0.7 ABCD	*1.1*
C33	2.9 CDEFGH	*2.6*	3.0 EFGHIJK	*2.5*
C34	5.7 JKLM	*3.1*	4.9 KLMNOP	*3.2*
C35	0.8 ABC	*1.1*	0.7 ABCD	*1.2*
C36	0.3 A	*0.5*	0.6 AB	*1.1*
C37	4.7 GHIJK	*3.2*	5.5 MNOPQR	*3.2*
C40	1.9 ABCDE	*2.7*	3.0 EFGHIJK	*3.3*

Table 3.4: *Mean scores, standard deviations and homogeneous subsets obtained through rating of the 40-model skin creams by the trained panel for the attributes slipperiness, absorption and final greasiness. Samples with the same letter, within a column are not significantly different to each other (p > 0.05).*

Cream	SLIPPERINESS		ABSORPTION		FINAL GREASINESS	
	Mean & Tukey's homogeneous subsets	***SD***	**Mean & Tukey's homogeneous subsets**	***SD***	**Mean & Tukey's homogeneous subsets**	***SD***
C1	6.7^{IJK}	*1.9*	7.7^{LM}	*2.8*	0.5^{A}	*0.8*
C2	9.3^{N}	*0.7*	7.4^{JKLM}	*3.2*	0.8^{A}	*1.3*
C3	8.9^{MN}	*1.2*	7.7^{KLM}	*3.2*	0.9^{A}	*2.0*
C3R	8.6^{MN}	*1.5*	7.6^{KLM}	*3.1*	0.9^{A}	*1.6*
C4	5.8^{HIJ}	*2.2*	1.7^{ABCD}	*2.6*	8.3^{KL}	*1.5*
C4R	5.6^{HI}	*2.1*	1.5^{ABC}	*2.1*	7.9^{KL}	*2.5*
C5	1.0^{A}	*0.8*	4.1^{FGH}	*2.9*	3.7^{CDEF}	*2.9*
C5R	1.9^{AB}	*1.9*	5.1^{GHI}	*3.0*	3.6^{BCD}	*2.4*
C6	8.7^{MN}	*1.1*	6.5^{IJKLM}	*2.6*	2.0^{ABC}	*2.8*
C7	4.3^{EFG}	*2.0*	2.5^{ABCDEF}	*1.8*	5.5^{EFGHI}	*2.2*
C7R	4.9^{FGH}	*1.7*	3.6^{DEFG}	*2.6*	5.3^{DEFGH}	*2.5*
C8	8.7^{MN}	*1.1*	7.0^{IJKLM}	*2.8*	1.2^{A}	*2.2*
C8R	8.9^{MN}	*1.0*	7.1^{IJKLM}	*2.7*	1.5^{A}	*2.9*
C9	4.2^{DEFG}	*2.2*	4.2^{FGH}	*2.8*	3.5^{BCD}	*2.1*
C10	9.1^{MN}	*0.7*	6.6^{IJKLM}	*3.2*	0.7^{A}	*1.6*
C11	8.9^{MN}	*0.9*	3.9^{EFGH}	*3.4*	5.1^{DEFG}	*3.4*
C11R	9.2^{MN}	*0.9*	2.6^{BCDEF}	*2.9*	5.6^{FGHIJ}	*3.0*
C12	4.2^{DEFG}	*2.2*	7.0^{IJKLM}	*3.2*	0.9^{A}	*1.3*
C12R	5.0^{FGH}	*2.2*	7.7^{KLM}	*3.0*	1.0^{A}	*1.8*
C14	7.9^{KLM}	*1.5*	1.8^{ABCD}	*2.7*	8.0^{KL}	*2.1*
C15	2.9^{BCD}	*1.8*	2.9^{CDEF}	*2.3*	5.2^{DEFGH}	*2.6*
C16	5.5^{GHI}	*2.1*	2.3^{ABCDEF}	*3.0*	7.2^{IJK}	*2.2*
C17	3.9^{DEF}	*2.1*	2.0^{ABCDE}	*2.3*	7.0^{HIJK}	*2.0*
C18	4.8^{FGH}	*1.9*	5.4^{GHIJ}	*2.9*	3.9^{CDEF}	*2.7*
C20	6.8^{IJK}	*1.4*	8.1^{M}	*3.0*	0.7^{A}	*1.2*
C23	3.9^{DEF}	*2.4*	5.7^{HIJKL}	*3.0*	4.8^{DEFG}	*2.9*
C24	3.3^{CDE}	*1.8*	5.7^{HIJK}	*3.1*	3.7^{CDE}	*2.5*
C25	8.4^{LMN}	*1.3*	1.8^{ABCD}	*2.6*	7.5^{JKL}	*2.6*
C27	1.5^{A}	*1.3*	8.0^{M}	*2.3*	0.8^{A}	*1.2*
C28	8.9^{MN}	*1.1*	0.6^{AB}	*1.2*	9.2^{L}	*1.2*
C29	7.9^{KLM}	*1.4*	7.7^{KLM}	*2.9*	1.3^{A}	*2.4*
C30	2.2^{ABC}	*1.9*	6.8^{IJKLM}	*3.4*	0.5^{A}	*1.1*
C31	9.0^{MN}	*1.4*	7.1^{IJKLM}	*3.2*	0.8^{A}	*2.0*
C32	7.1^{JKL}	*1.8*	1.6^{ABCD}	*2.7*	7.7^{KL}	*2.6*
C33	5.0^{FGH}	*2.0*	6.7^{IJKLM}	*2.5*	1.8^{AB}	*2.2*
C34	6.8^{IJK}	*2.1*	7.8^{M}	*2.2*	0.2^{A}	*0.3*
C35	8.3^{LMN}	*2.0*	1.0^{ABC}	*1.8*	8.1^{KL}	*1.9*
C36	9.1^{MN}	*0.8*	0.5^{A}	*1.0*	9.2^{L}	*1.2*
C37	8.5^{MN}	*1.3*	6.9^{IJKLM}	*2.7*	0.9^{A}	*1.7*
C40	1.8^{AB}	*1.6*	1.5^{ABC}	*1.6*	6.4^{GHIJK}	*2.8*

As observed in the preliminary rating of eight model skin creams (see Chapter 2.3.2.5), some panellists found it more difficult to discriminate between creams for certain attributes. These more challenging attributes included stickiness, cooling and drying.

The attributes stickiness and cooling appeared to be the most challenging from a QDA rating perspective. For the attribute stickiness, the panellist-product interaction plot showed a high level of cross-over which suggests panellists were struggling with the rating of this attribute. However, for some samples there was still agreement and a good range of the rating scale had been used, so it was decided that this attribute should be included in further analysis. On the other hand, results for cooling were not discriminating therefore these results were removed from any further analysis.

Panellist F was rating creams for the attribute drying in a different rank order to the other panellists and the range of the scale used by this panellist was mainly between 3 and 7, which is the mid range of the scale (see Figure 3.1). Results for this panellist were therefore removed from the data set for the attribute drying to prepare the results for further analysis. For the attribute final greasiness, panellist G was rating samples in a different rank order to the rest of the panel so this panellist's data for final greasiness was also removed from the final data set. Thus results given in Tables 3.3 and 3.4 exclude panellist F's drying data and panellist G's final greasiness data. Note that for the other attributes rating scores for panellist F and panellist G were in line with the other panellists, therefore their data was kept in further analysis of these attributes. It was thought that their different rating scores for these attributes was due to them having more hair on the back of their hands compared to the other panellists, this can affect the rating (perception) of attributes such as final greasiness and drying.

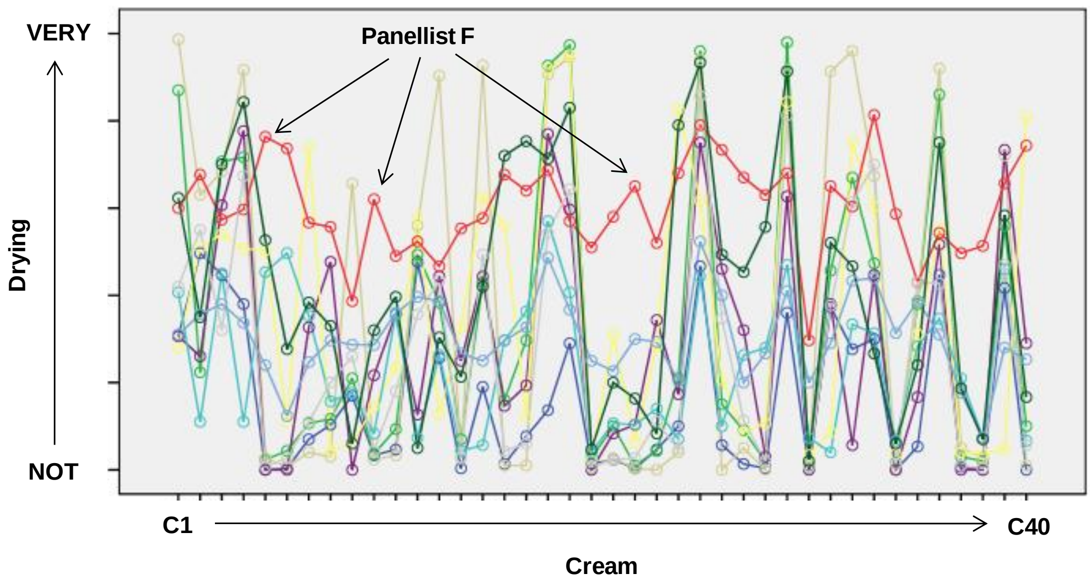


Figure 3.1: *Interaction plot showing average panellist scores for the attribute 'Drying'. Panellist F (red data points) was removed from further analysis due to different use of the rating scale.*

3.1.2 Relationships between textural attributes of model skin creams

PCA showed that the majority of variation within the data (91 %) could be explained by two axes, see correlation circle Figure 3.2. Principal component 1 (PC1) explained 53 % of the variation in the results. Attributes on this axis were all related to **initial application procedures** (see attribute definitions Chapter 2.3.2.3, Table 2.3). PC1 was positively correlated to the attributes firmness, thickness, resistance and spreadability (r = 0.96 - 0.99) and negatively correlated to slipperiness (r = -0.96). Consequently these attributes are correlated to each other. Thus if a sample is slippery it is unlikely to be firm, thick, have high resistance and be difficult to spread. Note that not all attributes had the same rating scale anchors. For the attribute spreadability, the rating scale went from easy to difficult therefore a high result for the attribute spreadability meant that the sample was difficult to spread (rather than being very spreadable as one might expect), see attribute definitions Chapter 2.3.2.3, Table 2.3. The scale anchors and rating protocols were chosen by the panel as is the case with QDA (see Chapter 1.4.1.1).

The attribute stickiness was only correlated by 0.79 with PC1 suggesting a slightly different relationship exists between stickiness and attributes on PC1. Principal component 2 (PC2) explained 38 % of the variation in results with positive correlation above 0.9 for drying, dragging and absorption (0.95 - 0.96) and negative correlation of -0.95 for final greasiness. Attributes correlated to this axis are related to interaction of the cream with the skin (**secondary application procedures**). PC2 indicates that if a sample absorbs quickly, is drying and dragging, it is unlikely to be greasy following application. The correlation circle in Figure 3.2 forms the foundation for further analysis presented in Chapter 4 whereby PCA and predictive modelling were used to understand correlations between sensory data and physical parameters.

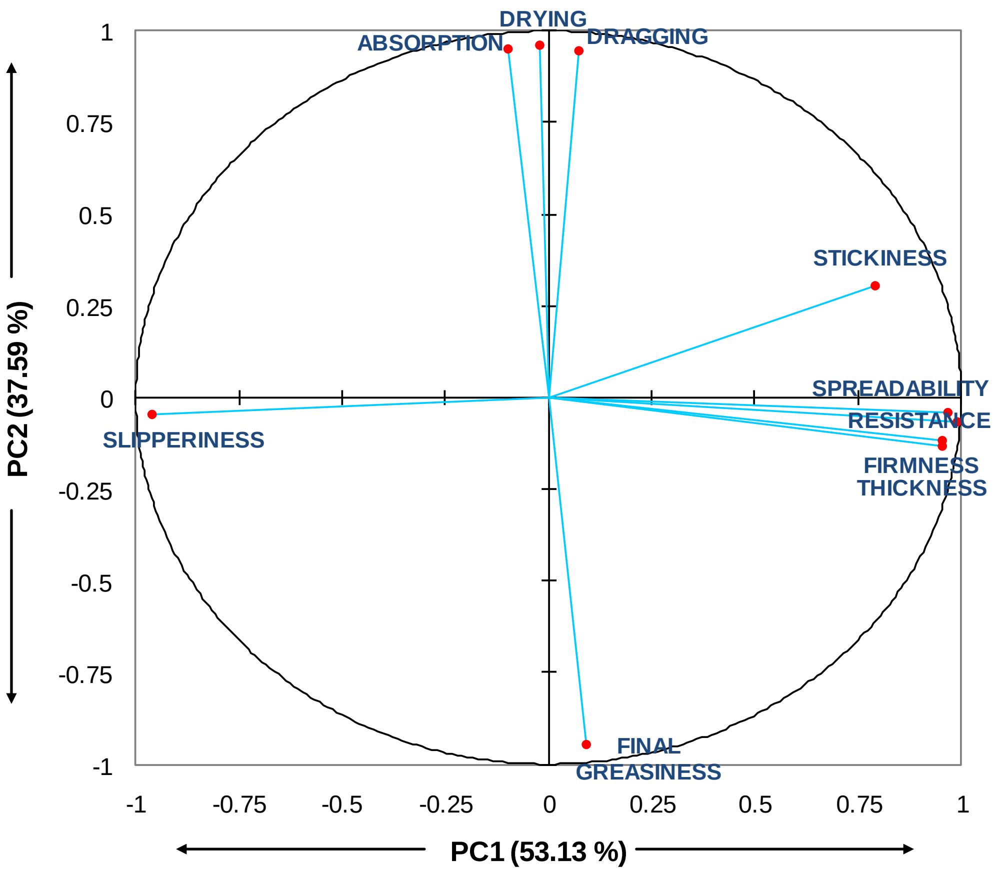


Figure 3.2: *PCA correlation circle illustrating the relationship between attributes rated by the trained panel (when rating 40 model skin creams).*

PC3, which explained 6 % of variation in the results, was also considered during analysis. However, a closer look at the correlation of attributes to PC1, PC2 and PC3 revealed that all sensory attributes were more highly correlated to PC1 and PC2; therefore PC3 has not been used in further analysis.

PCA biplots highlighted similarities and differences between creams, see Figure 3.3. Samples were located in all four quadrants of the PCA biplot illustrating the wide range of properties encompassed by the model skin creams. Significant differences were present between creams in the different quadrants for the textural attributes on PC1 and 2 ($p < 0.05$) and between certain creams within individual quadrants for the attribute firmness ($p < 0.05$). Similar relationships were found for other attributes on PC1 showing high discrimination ability. Stickiness, however, was an exception with few significant differences between creams in individual quadrants, although as already mentioned it had lower correlation with PC1 therefore the relationships are likely to differ. For the attribute final greasiness few significant differences were present between creams in the top two quadrants whereas some samples within the bottom two quadrants showed significant differences between them. The opposite relationship was observed for attributes positively correlated to PC2 i.e. few significant differences were observed between creams in the bottom two quadrants while significant differences were mainly present between creams in the top two quadrants (see Tables 3.1 - 3.4).

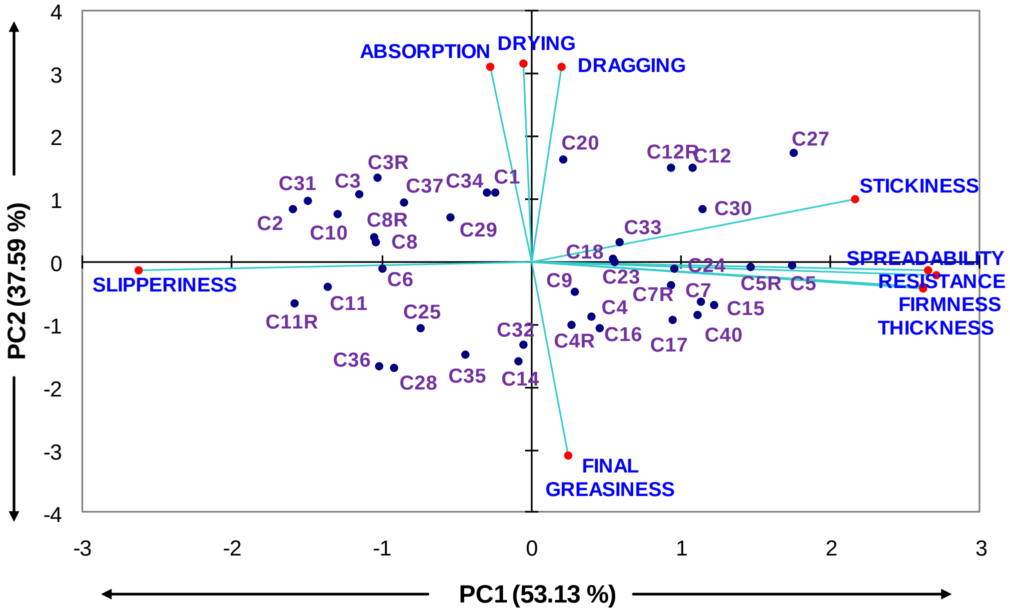


Figure 3.3: *PCA biplot showing the relationship between model skin cream samples and sensory attributes rated by the trained panel.*

Replicate samples were close together on the biplot indicating consistency amongst panellist rating and good batch to batch reproducibility of the skin cream production method. Looking at the ingredient composition of the skin creams (see Table 2.2, Chapter 2.2.1) it was clear that the firmer, thicker creams (those on the right of PC1) contained higher levels of SA (e.g. C5). Samples with low levels of SA were found to be more slippery (e.g. C2). This was expected since one role of the SA is to add body to the formulation (Eccleston, 1997). Parente et al. (2008) found a similar relationship when investigating emollients with different solids content, samples with higher levels of solids were found to be more difficult to spread and had a higher stickiness at skin temperature.

The relationship between oil content and skin cream properties was largely explained by PC2: creams containing no oil (e.g. C20) were more drying, dragging, absorbed quicker and had a lower final greasiness than those containing high levels of oil (e.g. C28). These properties are likely to be due to the high water content which evaporates during application allowing for easier application (Shai et al., 2001) hence the faster rate of absorption (see Chapter 1.3.4). The lack of oil leaves a non-greasy residue on the skin which explains the higher drying and dragging properties. These relationships are illustrated in Figures 3.4 and 3.5 respectively.

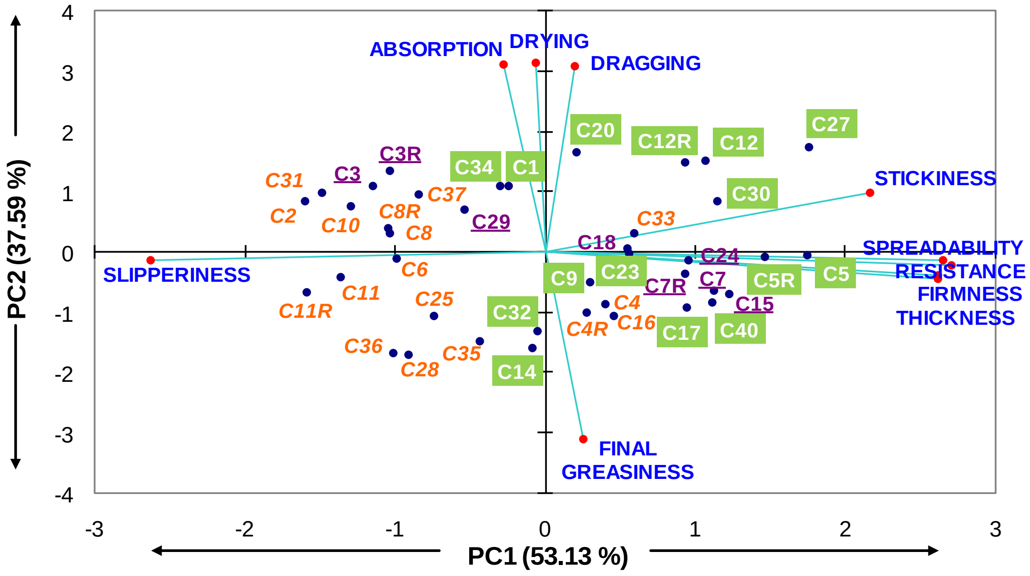


Figure 3.4: *PCA biplot showing the relationship between model skin cream samples and stearic acid (SA) level where **5 % SA**, **12.5 % SA**,* **20 % SA**

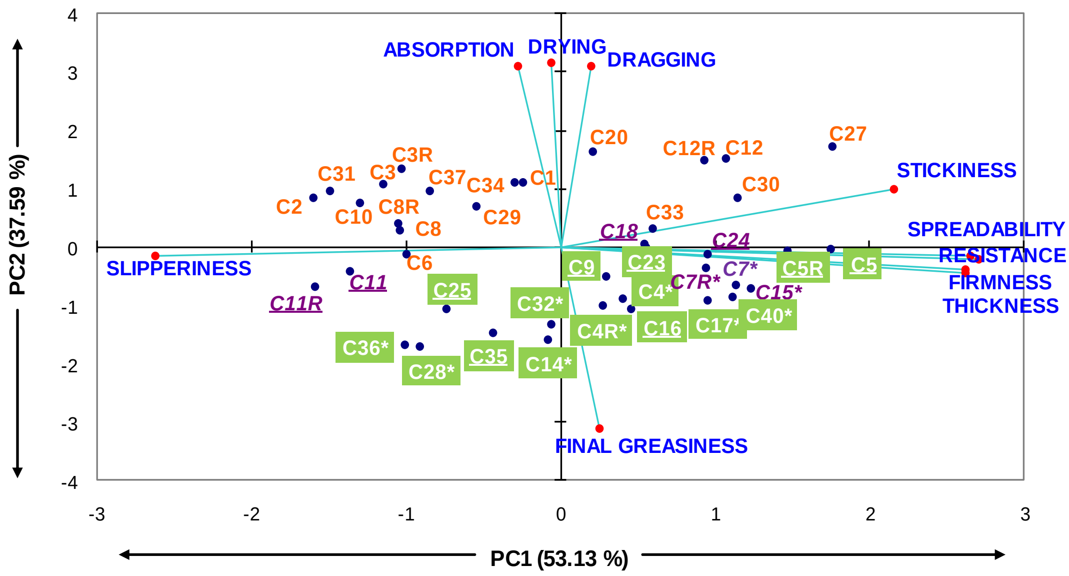


Figure 3.5: *PCA biplot showing the relationship between model skin cream samples and oil content where creams containing silicone oil are underlined, mineral oil is indicated by *, **no oil**, **20 % oil**,* **40 % oil**

More complex relationships were found for the other ingredient types and levels, however, these are not discussed because it is not the ingredient composition alone that affects the sensory properties. It is in fact the microstructure formed during skin cream production that determines the resulting sensory characteristics of creams (Wibowo and Ng, 2001). Although relationships between ingredients and resulting sensory properties in this model system can be observed, modifications to the skin cream manufacture procedure would allow production of creams with different sensory properties (i.e. microstructures) based on the same formulation. For example, rapid cooling of skin creams during manufacture results in a different microstructure than slow cooling, the amount of shear applied during the cooling stages is also important in determining the final microstructure (Eccleston, 1997; Telford, 2007).

The focus of this PhD was to understand how physical properties of skin creams relate to sensory attributes and ultimately how this in turn affects consumer liking. This research therefore attempts to correlate physical properties of skin creams with sensory properties, which could be applicable to a wide range of skin creams (see Chapter 4). On the other hand the ingredient relationship, although interesting, was only relevant to the creams used in this project produced using the manufacture procedure outlined in Chapter 2.2.2 hence it has not been discussed in detail.

3.1.3 Summary

40 model skin creams were rated in triplicate by the UoN trained panel for sensory (textural) attributes previously determined by QDA. Correlation circles revealed interesting relationships between attributes. Attributes relating to initial cream application procedures (firmness, thickness, resistance, spreadability, slipperiness) were highly correlated to PC1. Stickiness was an exception being weakly correlated to PC1 ($r = 0.79$). Attributes describing skin cream application

procedures involving absorption of cream into the skin i.e. secondary application procedures (drying, dragging, absorption, final greasiness) were correlated to PC2. Investigating these correlations between sensory attributes was important for further analysis in which physical parameters best describing sensory attributes on PC1 and 2 were selected for creation of predictive models (see Chapter 4).

3.2 UNDERSTANDING CONSUMER LIKING BEHAVIOUR

3.2.1 Skin cream selection

Figure 3.6 illustrates the differences in creams selected for the consumer study by highlighting their position on the PCA biplot developed from QDA of 40 model skin creams (see Table 2.2, Chapter 2.2.1 for composition). Three creams were chosen from each quadrant to maximise variety of the samples tested. These creams were freshly manufactured for this study.

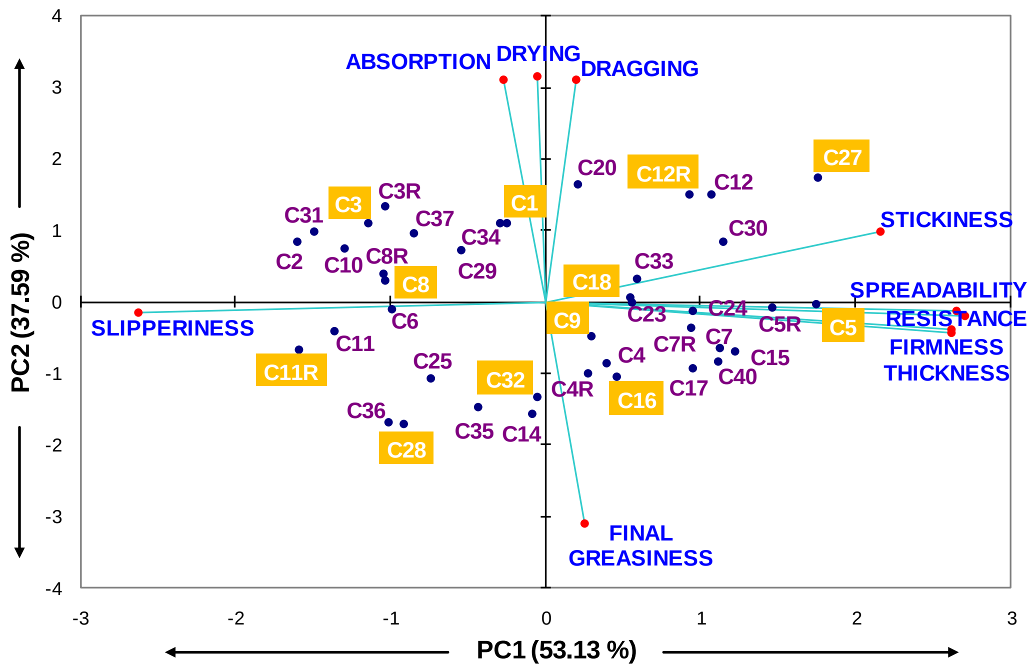


Figure 3.6: *PCA biplot showing the 12 creams selected for the consumer study as highlighted in orange.*

3.2.2 Textural properties of creams selected for consumer study

QDA results were analysed as described in Chapters 2.3.2.8. Two-way ANOVA revealed significant panellist, product and panellist-product interactions ($p < 0.05$), which as explained in Chapter 3.1.1 is common in sensory science even with trained panels. Taking into account the sample set, which included creams with a wide variety of sensory properties, these cross-over interactions were considered to be at an acceptable level.

Tukey's HSD post hoc test revealed that panellists were able to differentiate between the 12 skin creams for the different attributes. Panellist F's data was removed for the attributes drying and dragging due to different scale usage to that of other panellists (see also Chapter 3.1.1). The largest number of homogeneous subsets panellists separated samples into was 8 for the attribute stickiness (out of a maximum of 12). Results are summarised in Tables 3.5 - 3.7 where for each attribute mean panel scores, standard deviations and Tukey's homogeneous subsets are given.

Table 3.5: *Mean scores, homogeneous subsets and SD following rating of the 12 creams in triplicate for the attributes firmness, thickness, resistance and spreadability. Samples with the same letter, within a column are not significantly different to each other (p > 0.05).*

Cream	FIRMNESS		THICKNESS		RESISTANCE		SPREADABILITY	
	Mean & Tukey's homogen-eous subsets	***SD***	**Mean & Tukey's homogen - eous subsets**	***SD***	**Mean & Tukey's homogen-eous subsets**	***SD***	**Mean & Tukey's homogen-eous subsets**	***SD***
C1	3.9 B	*2.0*	4.8 B	*2.5*	3.1 C	*2.2*	2.7 C	*1.2*
C3	0.8 A	*1.0*	1.3 A	*1.0*	0.4 A	*0.3*	0.3 A	*0.3*
C5	9.5 E	*0.5*	9.6 E	*0.4*	9.0 F	*0.7*	8.2 E	*1.3*
C8	3.8 B	*1.8*	4.4 B	*2.3*	1.6 B	*1.6*	1.3 AB	*1.1*
C9	5.8 C	*1.9*	6.3 CD	*2.1*	5.0 D	*2.2*	4.6 D	*1.8*
C11R	0.1 A	*0.1*	0.4 A	*0.5*	0.3 A	*0.5*	0.2 A	*0.3*
C12R	8.6 E	*1.3*	8.7 E	*1.0*	6.4 E	*2.4*	4.8 D	*2.5*
C16	6.9 D	*1.6*	7.4 D	*1.7*	5.3 DE	*1.8*	3.9 D	*2.2*
C18	5.9 CD	*1.9*	6.2 C	*2.1*	4.5 D	*2.0*	2.3 BC	*1.9*
C27	9.1 E	*2.0*	9.6 E	*0.4*	9.0 F	*1.1*	8.5 E	*1.3*
C28	3.9 B	*2.2*	3.9 B	*2.4*	1.0 AB	*0.9*	1.0 A	*0.8*
C32	6.3 CD	*2.1*	6.8 CD	*2.3*	4.6 D	*2.6*	4.4 D	*2.1*

Table 3.6: *Mean scores, standard deviations and homogeneous subsets obtained through rating the 12 creams in triplicate for the attributes stickiness, drying and dragging. Samples with the same letter, within a column are not significantly different to each other (p > 0.05).*

Cream	STICKINESS		DRYING		DRAGGING	
	Mean & Tukey's homogeneous subsets	***SD***	**Mean & Tukey's homogeneous subsets**	***SD***	**Mean & Tukey's homogeneous subsets**	***SD***
C1	3.8 ABCD	*2.7*	6.3 F	*3.1*	5.6 CD	*3.6*
C3	4.7 CDE	*3.0*	7.4 F	*2.7*	7.7 E	*2.8*
C5	6.9 FGH	*2.7*	1.7 BCD	*1.4*	2.4 B	*2.0*
C8	4.6 BCDE	*2.8*	4.8 E	*3.5*	4.3 C	*3.7*
C9	5.4 DEF	*2.8*	1.6 ABC	*1.5*	1.6 AB	*1.5*
C11R	3.1 ABC	*3.0*	2.5 CD	*3.1*	2.1 AB	*2.6*
C12R	8.3 GH	*1.3*	7.3 F	*2.7*	7.6 E	*2.0*
C16	6.5 FG	*1.7*	0.5 AB	*0.6*	0.7 A	*1.1*
C18	6.1 EF	*2.4*	3.0 D	*3.0*	2.5 B	*2.6*
C27	8.4 H	*1. 2*	6.5 F	*2.6*	6.7 DE	*2.7*
C28	2.8 A	*2.5*	0.3 A	*0.4*	0.7 A	*1.2*
C32	2.9 AB	*2.6*	0.5 AB	*0.7*	1.0 AB	*1.5*

Table 3.7: *Mean scores, standard deviations and homogeneous subsets obtained through rating the 12 creams in triplicate for the attributes slipperiness, absorption and final greasiness. Samples with the same letter, within a column are not significantly different to each other (p > 0.05).*

Cream	SLIPERINESS		ABSORPTION		FINAL GREASINESS	
	Mean & Tukey's homogeneous subsets	***SD***	**Mean & Tukey's homogeneous subsets**	***SD***	**Mean & Tukey's homogeneous subsets**	***SD***
C1	6.7 CD	*2.2*	5.1 BC	*1.9*	0.9 AB	*1.5*
C3	8.5 EF	*1.5*	3.1 A	*1.4*	0.4 A	*0.9*
C5	2.2 A	*2.3*	7.9 EF	*2.2*	4.6 C	*2.8*
C8	8.6 F	*1.1*	6.6 CDE	*1.6*	2.1 B	*2.8*
C9	5.1 B	*2.3*	7.0 DE	*2.5*	4.9 C	*2.6*
C11R	9.1 F	*1.1*	4.8 AB	*3.1*	5.0 C	*3.4*
C12R	5.7 BC	*2.4*	5.1 BC	*1.5*	1.1 AB	*1.4*
C16	5.9 BC	*1.5*	9.1 FG	*1.4*	7.2 D	*1.8*
C18	6.1 BC	*1.8*	6.6 CDE	*2.5*	4.4 C	*2.9*
C27	2.0 A	*2.0*	6.1 BCD	*2.7*	1.2 AB	*1.6*
C28	9.2 F	*0.7*	9.9 G	*0.1*	9.1 E	*1.0*
C32	7.4 DE	*1.7*	9.7 G	*0.5*	7.9 DE	*1.8*

Overall average results showed excellent agreement with data obtained from rating the 40 creams in triplicate, see Figures 3.7 and 3.8 which compare the average results for 'firmness' (PC1 attribute) and 'final greasiness' (PC2 attribute) respectively.

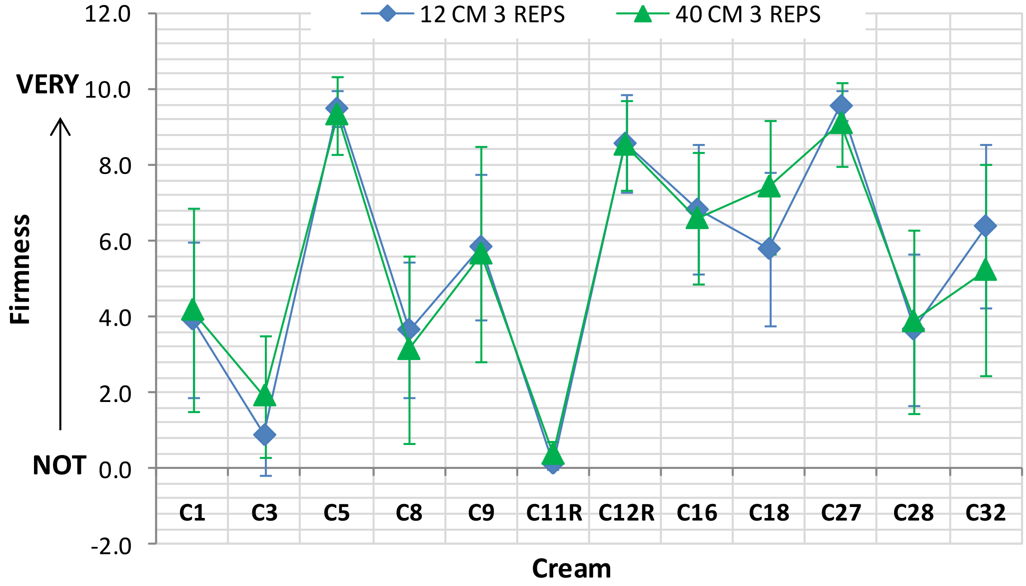


Figure 3.7: *Average firmness scores as rated by the trained panel for the 40 model skin creams (40cm 3 reps) and the 12 creams used in the consumer study (12 cm 3 reps).*

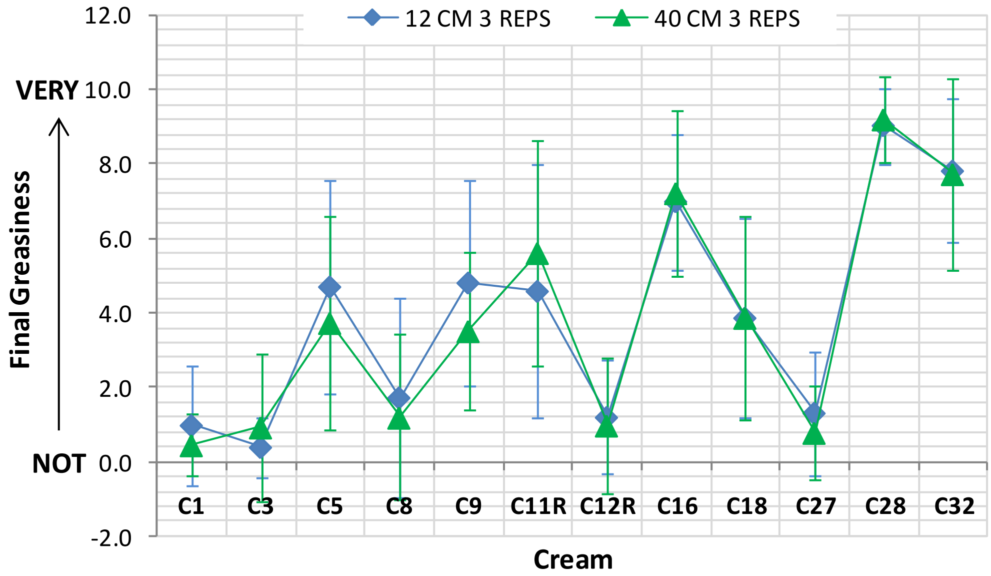


Figure 3.8: *Average final greasiness scores as rated by the trained panel for the 40 model skin creams (40cm 3 reps) and the 12 creams used in the consumer study (12 cm 3 reps).*

PCA was also performed on trained panel results from rating of the 12 creams in triplicate. The result given in Figure 3.9 was very similar to that obtained for the 40 creams (see Figure 3.2, Chapter 3.1.2), in this case however, 'absorption' was negatively correlated to PC2 since the rating scale ends were reversed (from 'slow to fast' to 'fast to slow') when rating the 12 consumer study creams (as described in Chapter 2.3.3.2). The agreement between results from rating 40 creams 3 replicates and that of 12 creams 3 replicates indicates consistency in cream manufacture and panel rating.

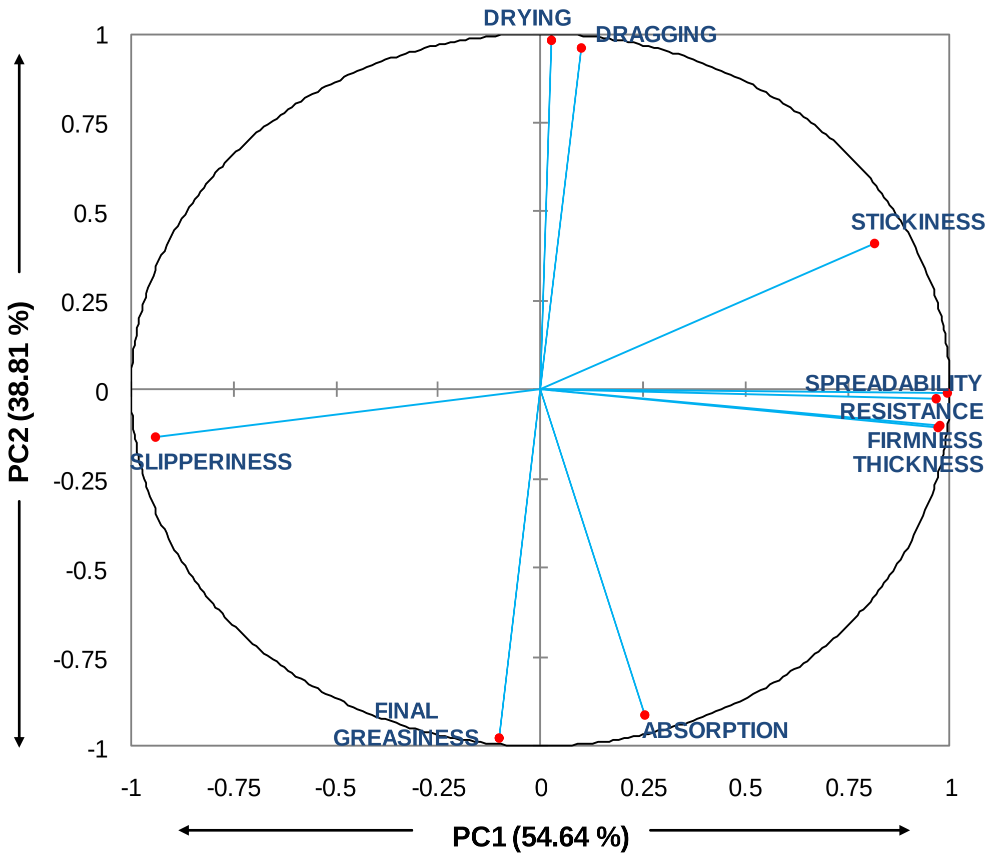


Figure 3.9: *PCA correlation circle illustrating the relationship between attributes rated by the trained panel (when rating 12 consumer study creams).*

3.2.3 Consumers' background

Of the 150 volunteers, 148 attended both sessions therefore data from the other two consumers was discarded. Questionnaire results provided good background information about the consumers involved in the study (see Appendix VI for a copy of the questionnaire). The majority of participants were female (72 %), aged between 16 and 60+ (see Figure 3.10) with a large number of 21-25 year olds (33 %). Most participants used hand cream on a regular basis; questionnaire results showed that 72 % of participants used hand cream once a day or more often (see Figure 3.11). Results also indicated that 50 % of volunteers buy products for normal skin, 39 % buy for dry skin and the remaining 11 % includes those who purchase for oily skin or other.

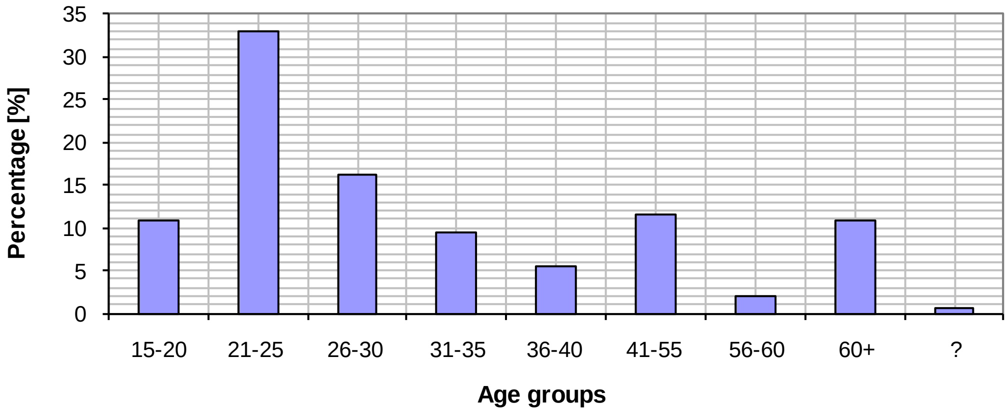


Figure 3.10: *Age ranges of participants in the hand cream consumer study.*

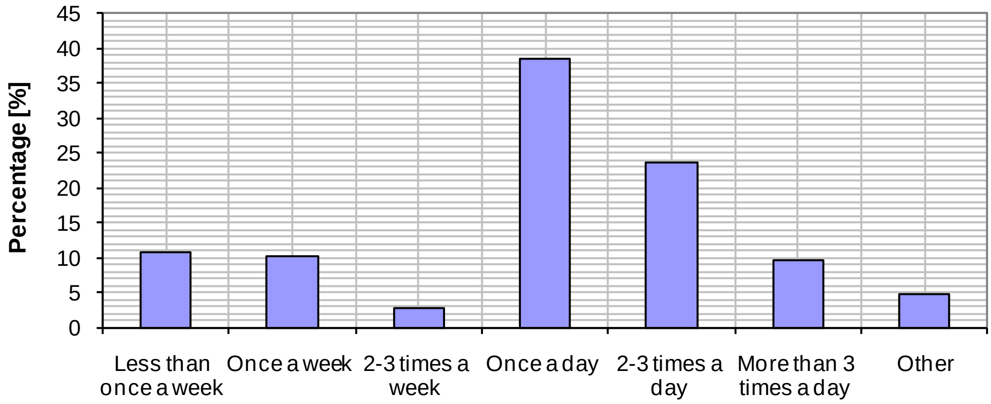


Figure 3.11: *Skin cream usage habits of consumers that participated in the hand cream consumer study.*

3.2.4 Agglomerative hierarchical clustering (AHC)

Agglomerative hierarchical clustering was carried out according to the methods described in Chapter 2.3.3.5. Three clusters were identified representing different types of consumer liking behaviour. Clusters one to three contained 47, 34 & 61 consumers respectively. Note that the total number of consumers in this Chapter is equal to 142 since preliminary AHC identified 6 outliers which were removed from further analysis, see Chapter 2.3.3.5. Average consumer liking scores for each cluster are presented in Figure 3.12. The y-axis represents the LAM scale used by the consumers (see Figure 2.9, Chapter 2.3.3.4) where 50 % is equivalent to ‘neither like nor dislike’, above 50 % relates to liking (100 % is the greatest imaginable like) and anything below 50 % relates to dislike (0 % is the greatest imaginable dislike).

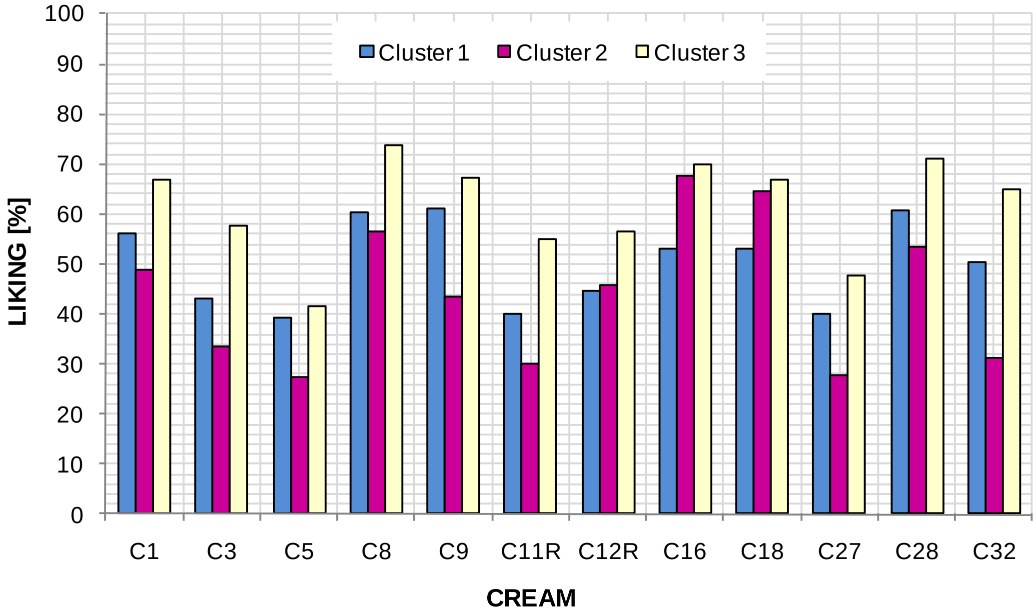


Figure 3.12: *Average consumer liking data for the three clusters obtained from cluster analysis.*

Considering the wide range of creams used in the consumer study it might be expected that a broader range of the scale would have been used. However, the results presented in Figure 3.12 are average results only and typically consumers avoid using the ends of scales (Kemp et al., 2009), which is why the majority of results were between 25 – 75 % liking. Also the samples used in this study were model skin creams therefore it was expected that liking scores of 0 % (greatest imaginable dislike) or 100 % (greatest imaginable like) would not arise.

Consumers in cluster one had mixed opinions on liking. Cluster two consumers were generally dissatisfied with the creams used in this study except for creams 8, 16, 18 and 28 which had overall properties (visual and textural) closest to those one might buy on the market compared to the other samples. Consumers in cluster three liked all samples except creams 5 and 27. Consumers in clusters one and three followed a similar pattern of liking behaviour except that cluster three generally scored creams higher than cluster one (see Figure 3.12). The liking scores for cluster two were generally lower than clusters one and three for the majority of skin cream samples exceptions being creams 16 and 18 which were medium creams (not too thin or thick) with low drying and dragging properties. It was clear that all participants disliked creams 5 and 27. This is understandable as these samples were the thickest extremes (i.e. thicker than a standard cream one might buy).

In order to understand the differences between the three groups of consumers, questionnaire results for consumers in the three clusters were compared. Sensory expectations for skin care products are related to culture, age, skin type, gender, setting and climate (Van Reeth, 2006), see Chapter 1.4.2. Therefore, it was thought that these factors may also affect consumer liking. However, in this case no firm correlations between questionnaire results and the three cluster groups were found. This suggests that for the cream samples used in this study, liking does not depend on age, gender, skin cream usage habits or skin type. On the other hand when consumers purchase products, marketing and brand image plays a huge role in consumer choice as consumers seek to purchase products that will suit their skin type and fit in with their image. For example male consumers are likely to purchase creams marketed as ‘for men’ or packaged in less feminine style packaging, to fit in with their masculine image.

3.2.4.1 Relationship between consumer liking and sensory properties of cream

Consumer liking behaviour and sensory properties of the skin creams were analysed to see whether different sensory properties were driving consumer liking in the three clusters. Extreme cream samples regarding attributes on PC1 (see Figures 3.6 and 3.9) were generally disliked by all clusters although the extent to which they were acceptable differed between clusters. Overall a broader range of sensory properties was acceptable to cluster three than to clusters one and two (see Figures 3.13 and 3.14 which show the QDA sensory rating scores for creams liked and disliked by cluster three).

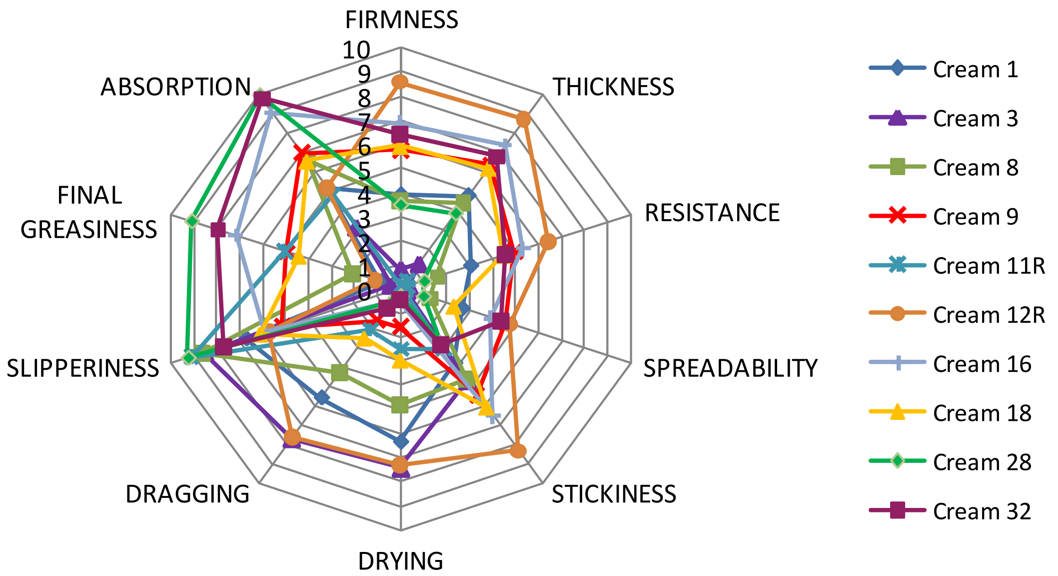


Figure 3.13: *Sensory properties of creams liked by cluster three.*

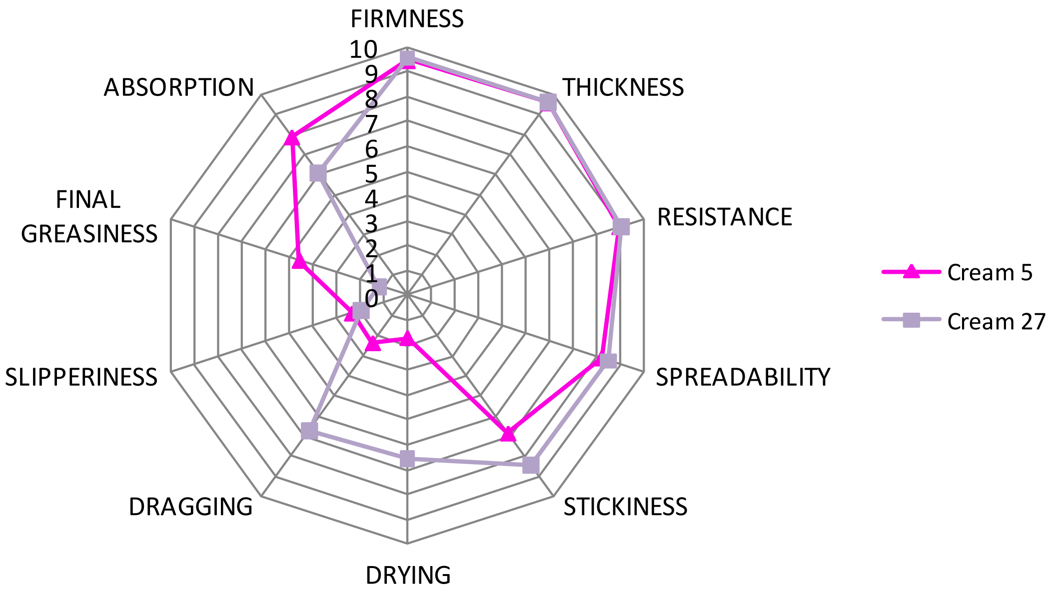


Figure 3.14: *Sensory properties of creams disliked by cluster three.*

Firmness and thickness

Consumers in cluster one liked all samples with firmness and thickness QDA scores within the range 3 – 8; all creams outside this range were disliked by this cluster. The liking behaviour for consumers in cluster two was more complicated but it was clear that they too disliked extreme cream samples in terms of firmness and thickness (scores outside the range 3 – 8). On the other hand, consumers in cluster three liked all samples with firmness and thickness scores of less than 9. These observations suggest that hand creams[1] with a wide range of firmness and thickness properties were acceptable to most consumers (ranges 3 – 8, cluster one or 0 – 9 cluster three). The creams that were unacceptable were extreme cream samples thus much thicker and firmer (or thinner and less firm) than common products found on the market.

Consumers in cluster three appeared to be unaffected by samples towards the thinner extreme, suggesting that extremely thin samples were more acceptable than extremely thick samples. This could be related to the fact that a runny sample is more likely to absorb quickly compared to a really thick sample. In his work carried out on lotions, Braun (1991) found that thicker samples (η > 2000 mPa s measured using a Brookfield viscometer with a spindle speed of 60 rpm; yield values > 50 Pa) were unacceptable to consumers as they absorbed too slowly, while samples of medium thickness (η = 1000 – 2000 mPa.s; yield values 25 – 50 Pa) were just acceptable. Samples with even lower viscosities absorbed at much faster rates. These findings relate to lotions therefore the viscosity boundaries differ in this study, however it is clear that samples of medium firmness and thickness were preferred, and the thinnest samples creams 3 and 11R absorbed at faster rates than the thickest samples creams 5 and 27 (see Table 3.7, Chapter 3.2.2). Although overall

[1] The term 'hand cream' is used here since participants in the consumer study were asked to rate their liking scores regarding the feel of the product as a hand cream base rather than a general skin cream.

the thickness was not directly proportional to the absorption rate since other factors such as the final greasiness of the sample also affected the absorption.

Resistance and spreadability

Consumers in all clusters disliked creams with very high resistance (≥ 9) and creams that were too difficult to spread (spreadability scores ≥ 8), consumers in cluster three liked all other samples whereas consumers in clusters one and two also disliked the other extreme (samples with very low resistance, < 1 and those that were very easy to spread, < 1). Consumers in clusters one and two only liked samples with resistance scores between 1 and 5.5. However, consumers in cluster two also disliked some samples within this range, as was the case with firmness and thickness (see Table 3.8). Interestingly, cream 12R although very thick (firmness and thickness scores 8.6 and 8.7 respectively) had resistance and spreadability scores much lower than creams 5 and 27 (the other thick samples) and it was more slippery. This could explain why it was liked by consumers in cluster three and preferred out of the other thick creams by consumers in clusters one and two.

Table 3.8: *QDA scores for consumer study creams, ten attributes, samples liked by cluster two are given in bold, purple font (creams 8, 16, 18 and 28).*

	C1	C3	C5	**C8**	C9	C11R	C12R	**C16**	**C18**	C27	**C28**	C32
FIRMNESS	3.9	0.8	9.5	**3.7**	5.8	0.1	8.6	**6.9**	**5.9**	9.6	**3.5**	6.4
THICKNESS	4.8	1.3	9.6	**4.4**	6.3	0.4	8.7	**7.4**	**6.2**	9.6	**3.9**	6.8
RESISTANCE	3.1	0.4	9	**1.6**	5	0.3	6.4	**5.3**	**4.5**	9	**1**	4.6
SPREAD-ABILITY	2.7	0.3	8.2	**1.3**	4.6	0.2	4.8	**3.9**	**2.3**	8.5	**1**	4.4
STICKINESS	3.8	4.7	6.9	**4.6**	5.4	3.1	8.3	**6.5**	**6.1**	8.4	**2.8**	2.9
DRYING	6.3	7.4	1.7	**4.8**	1.6	2.5	7.3	**0.5**	**3**	6.5	**0.3**	0.5
DRAGGING	5.6	7.7	2.4	**4.3**	1.6	2.1	7.6	**0.7**	**2.5**	6.7	**0.7**	1
SLIPPER-INESS	6.7	8.5	2.3	**8.6**	5.1	9.1	5.7	**5.9**	**6.1**	2	**9.3**	7.7
FINAL GREASINESS	0.9	0.4	4.6	**2.1**	4.9	5	1.1	**7.2**	**4.4**	1.2	**9.1**	7.9
ABSORPTION	5.1	3.1	7.9	**6.6**	7	4.8	5.1	**9.1**	**6.6**	6.1	**9.9**	9.7

Stickiness, drying, dragging, slipperiness, final greasiness and absorption

The stickiness, slipperiness, drying, dragging and final greasiness properties of the creams liked by the different clusters was more difficult to interpret. However, it appears that samples with slower absorption (> 6.5, note the scale ends go from fast to slow, see Chapter 2.3.3.2) were preferred. This trend only applies if the sample is within the acceptable firmness, thickness, resistance and spreadability ranges.

3.2.4.2 Summary of cluster analysis results

Overall cluster analysis revealed that consumer liking appears to be related to the sensory properties of skin creams as rated by the trained panel (external data) rather than skin type, cream usage habits or age (questionnaire results). In particular the extreme cream samples, creams 3, 5, 11R, 12R & 27 (e.g. very thick, not at all thick), were disliked by the consumers, see Figure 3.15. External preference mapping was therefore chosen for further analysis (rather than internal preference mapping) because it assumes that the subjects share a common perceptual space that can be defined by external data (MacFie, 2007) - in this case the attributes rated by the trained panel (see also Chapter 1.4.3.3 for further information on external preference mapping and Chapter 2.3.3.5 for the method used).

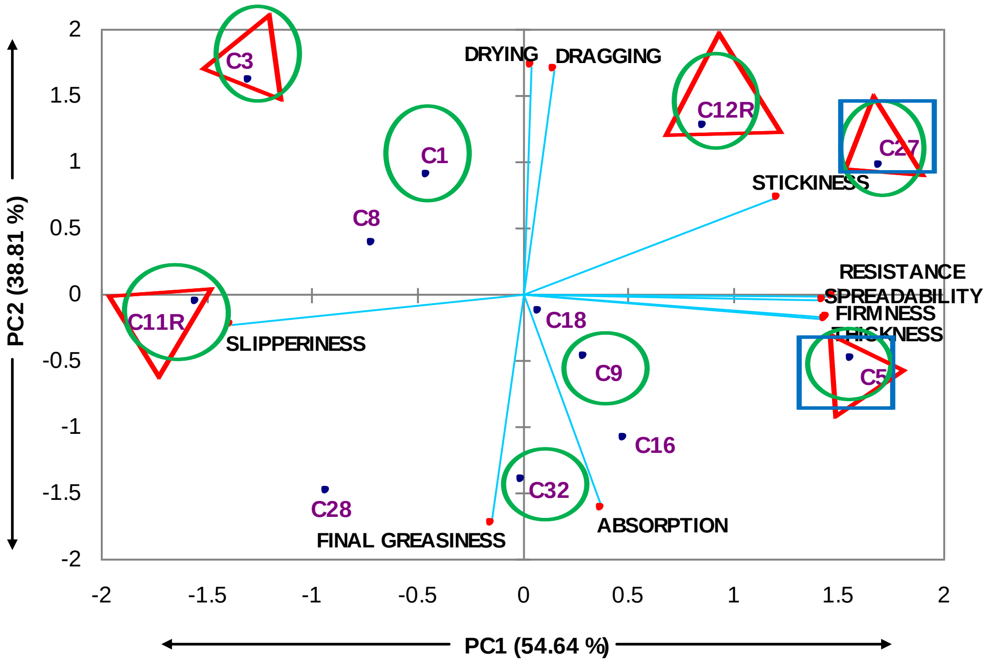


Figure 3.15: *PCA biplot highlighting creams disliked by cluster one (red triangles), cluster two (green circles) & cluster three (blue rectangles).*

3.2.5 External preference mapping

External preference mapping allows the relationship between consumer liking and sensory properties of creams to be understood in terms of models characterising different patterns of liking behaviour (see Chapter 1.4.3.3). Results revealed that the majority of consumers followed vector (103 consumers, 73 % of participants) or ideal point model trends (33 consumers, 23 % of participants) with just four saddle point models (3 %) and two anti-ideal point models (1 %). Note that the total number of consumers in this Chapter is equal to 142 since external preference mapping follows on from cluster analysis and preliminary AHC identified 6 outliers which were removed from further analysis, see Chapter 2.3.3.5 and Chapter 3.2.4. Less than 50 % of the consumer models showed significant fit (39 % significant fit where $p < 0.1$) but this is typical in external preference mapping (MacFie, 2010). Faber et al. (2003) suggested increasing the number of principal components to improve fit statistics but in this case the overall fit was not improved by this method. This is understandable as 93.45 % of variation in sensory attributes was explained by two principal components. Therefore it is unlikely that adding further components would improve the fit, although it is possible that another factor not measured could be driving liking. Initial analysis was followed by external preference mapping on the vector model and the non-vector model consumers separately to improve understanding of the results. Results will be discussed separately.

3.2.5.1 Vector models

Preference mapping revealed vector models spanning three quadrants of the design space suggesting that there were different patterns of liking amongst the consumers with vector models (see Figure 3.16). Cluster analysis was therefore performed on the liking data of consumers with vector models to explain these relationships (the Euclidean distance and Wards agglomeration method were used). Results revealed three clusters containing 40, 43 and 20 consumers respectively; interpretation of these results will be discussed separately.

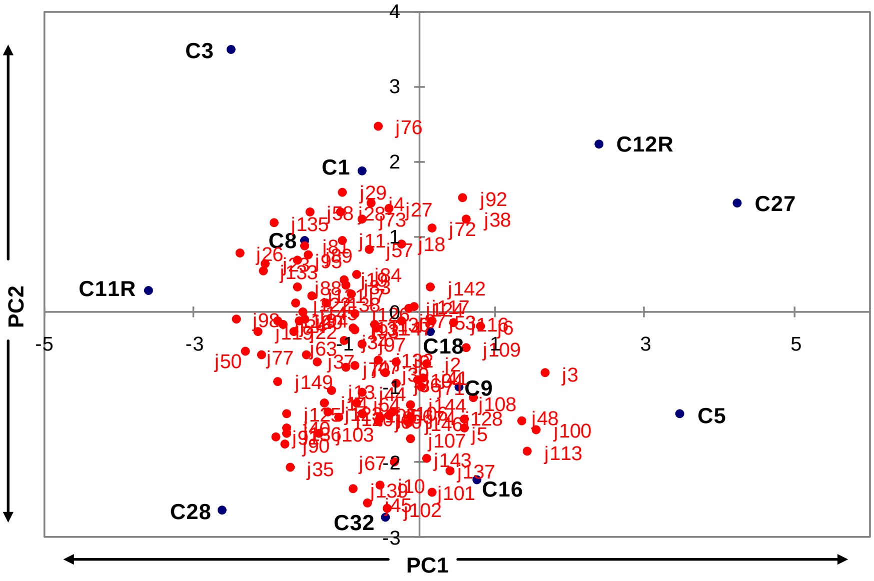


Figure 3.16: *External preference map for consumers with vector model liking trends.*

Consumer liking behaviour: Vector models, cluster one

The majority of vector models in cluster one were plotted on the negative axis of PC2 particularly in the direction of final greasiness and absorption (see Figure 3.17), suggesting as the final greasiness and absorption time increases (see Chapter 2.3.3.2), consumers like the samples more. There were some exceptions e.g. consumer 92 (j92) whose direction of liking was towards the opposite side of PC2. Further analysis of the sensory properties of creams liked by this cluster improved understanding of this relationship. Samples liked by more than 50 % of vector model consumers in cluster one were creams 5, 9, 11R, 16, 18, 28 and 32 (see Figure 3.18) all of which had final greasiness scores greater than 3 and absorption scores greater than 6.5 (cream 11R was an exception with an absorption of 4.8). This indicates that in general this cluster disliked samples that were not at all greasy following application and samples that absorbed rapidly, suggesting they liked a greasier feel on their skin.

As well as the final greasiness and absorption, the drying and dragging characteristics of these samples appeared to be driving liking which is not surprising as they are positively correlated to PC2 (if a sample is greasy it is unlikely to be drying or dragging, see Chapter 3.1.2). A small window of drying and dragging properties (QDA scores 0 - 3) was liked by more than 50 % of consumers, samples that were very drying and dragging were less acceptable to the majority of consumers (only 10 – 30 % of consumers liked samples with drying and dragging scores > 5). Figure 3.19 illustrates these relationships.

Skin creams are used to provide moisture to the skin and to prevent it from drying out (Shai et al., 2001; Kampf G. and Ennen J., 2006). Therefore it might be expected that skin creams will not be drying or dragging in nature which could explain why these consumers preferred samples that were not very drying or dragging. This in turn explains the result for final greasiness. If the skin was not at all greasy following application (or if it absorbed too quickly) it may be assumed that the product has not performed well i.e. it has not fulfilled its function in providing moisture to the skin.

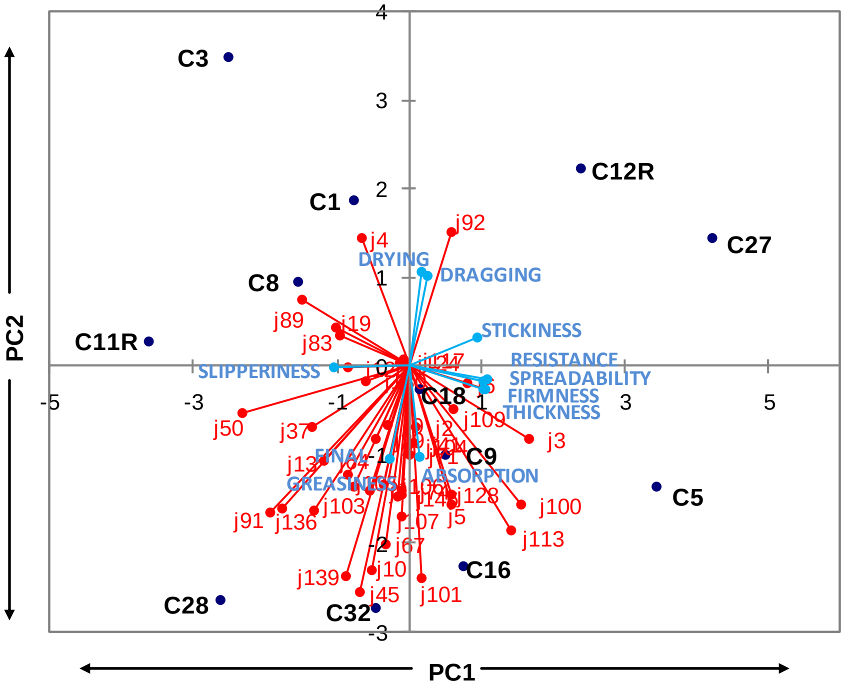


Figure 3.17: *Preference map for consumers with vector models in cluster one.*

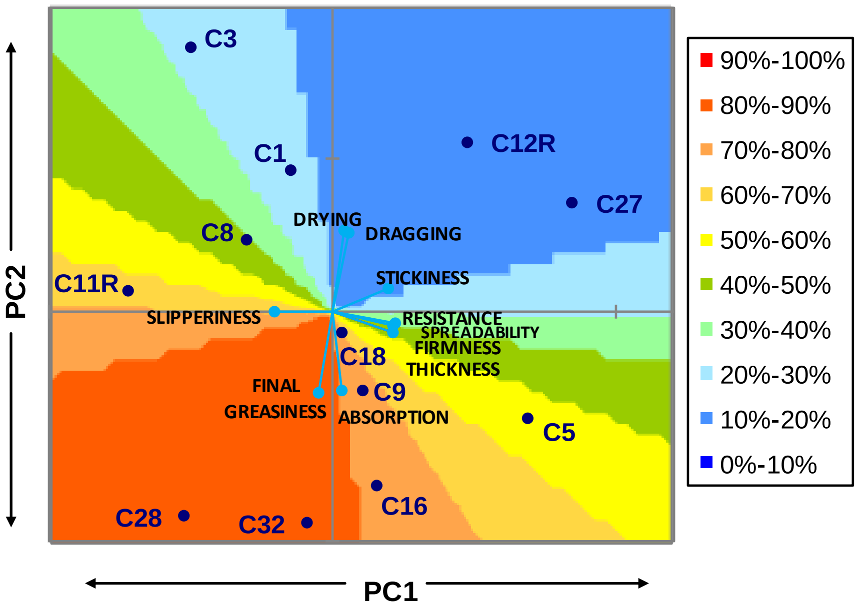


Figure 3.18: *Contour plot for consumers with vector models in cluster one.*

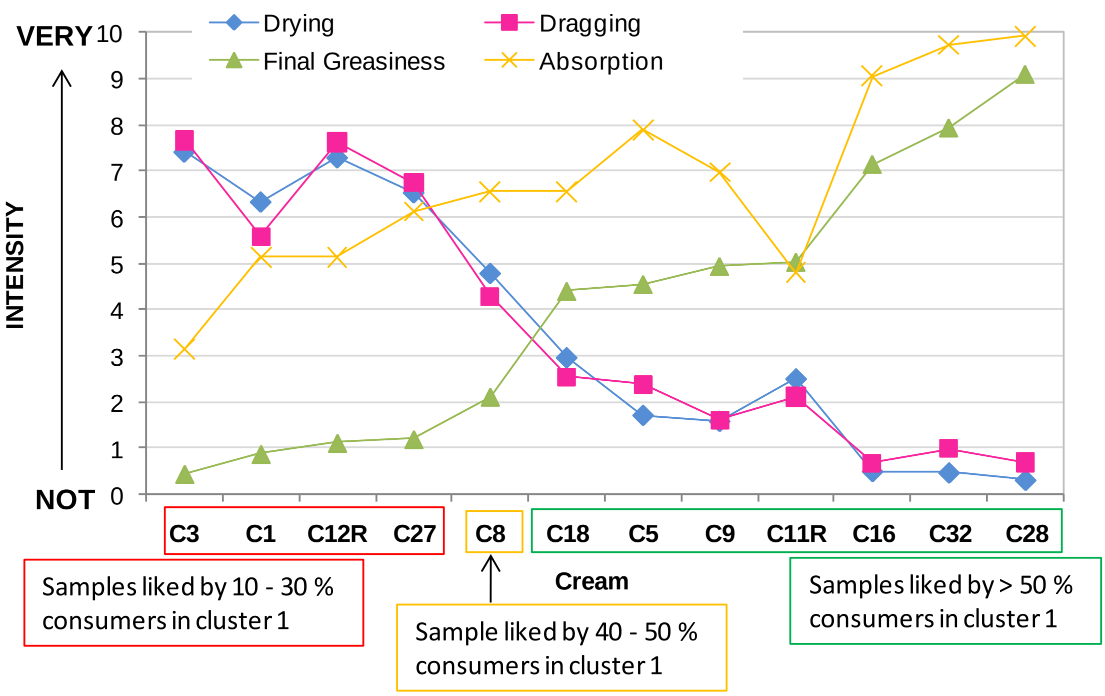


Figure 3.19: *QDA scores for the attributes on PC2 plotted in order of increasing final greasiness thus illustrating the effect these sensory properties have on consumer liking.*

Consumer liking behaviour: Vector models, cluster two

The majority of vector models in cluster two appear to be negatively correlated to PC1 in the direction of slipperiness (see Figure 3.20). Creams 8, 11R, 28 and 32 were liked by 70 – 90 % of vector model consumers in this cluster (see Figure 3.21). Looking at the sensory data for these samples showed that they were all very slippery (QDA score > 7) compared to the others[1]. Therefore in this case increasing the slipperiness appeared to increase liking for consumers in this cluster. However, the PCA correlation circle showed that increasing the slipperiness is correlated to a decrease in firmness, thickness, resistance, difficulty of spreading and to some extent stickiness (see Chapter 3.1.2). These attributes are more likely to be affecting consumer liking behaviour. Figure 3.22 shows the QDA scores for these attributes, plotted in order of increasing slipperiness. It indicates that the

[1] Cream 3 also had a high slipperiness (8.5), this sample was liked by 50 – 60 % consumers in this cluster.

relationship between firmness (and other attributes positively correlated to PC1) increasing and slipperiness decreasing was not linear. It was however apparent that creams liked by more than 50 % of consumers had spreadability scores < 5, resistance < 6, firmness < 8 and thickness < 7.

Looking at the preference map for cluster 2 (Figure 3.20) also shows a large number of consumers are in the bottom left quadrant suggesting that liking may also be related to the final greasiness. Final greasiness scores for creams liked by 70 – 90 % of consumers (creams 32, 8, 11R, 28) ranged between 2 and 9 indicating limited effect on liking. On the other hand the drying and dragging scores for these samples were all < 5 suggesting that consumers in cluster 2 preferred samples with low drying and dragging properties. Note that the effects drying or dragging properties have on liking depends on the slipperiness (and other attribute properties on PC1). If a sample is too firm or not at all slippery then it does not matter whether the sample has low drying or dragging properties as the dislike for high firmness or low slipperiness dominates and the sample is disliked. Cream 5 is a good example with low drying and dragging scores of 1.7 and 2.4 respectively, yet it has high scores for firmness (9.5), thickness (9.6), resistance (9.0), spreadability (8.2) and low slipperiness (2.2). Cream 5 was only liked by 20 – 30 % of consumers.

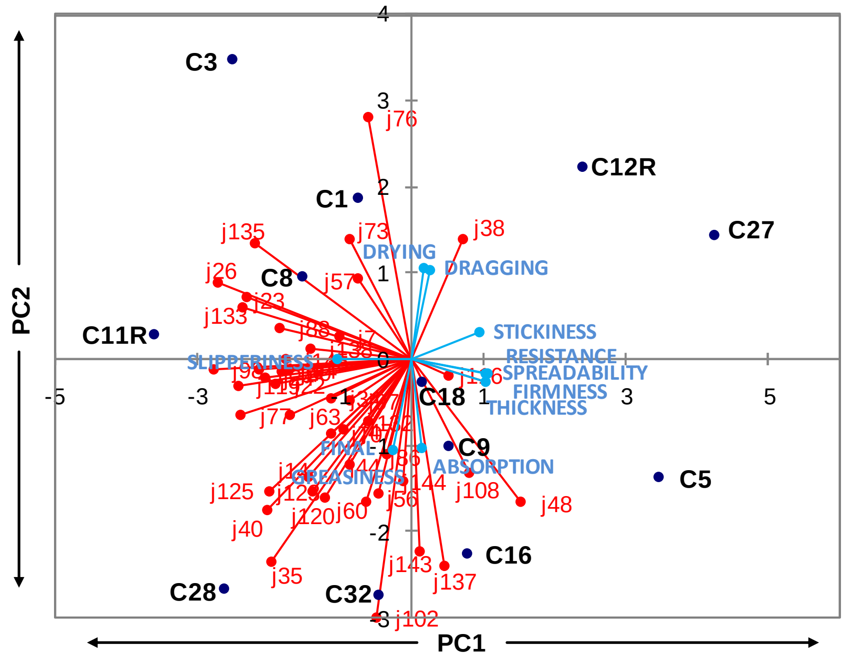


Figure 3.20: *Preference map for consumers with vector models in cluster two.*

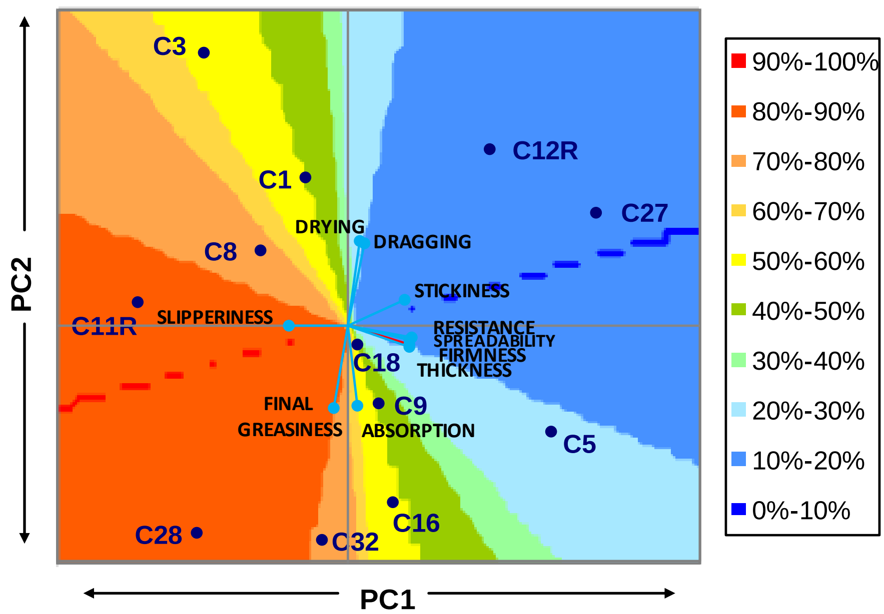


Figure 3.21: *Contour plot for consumers with vector models in cluster two.*

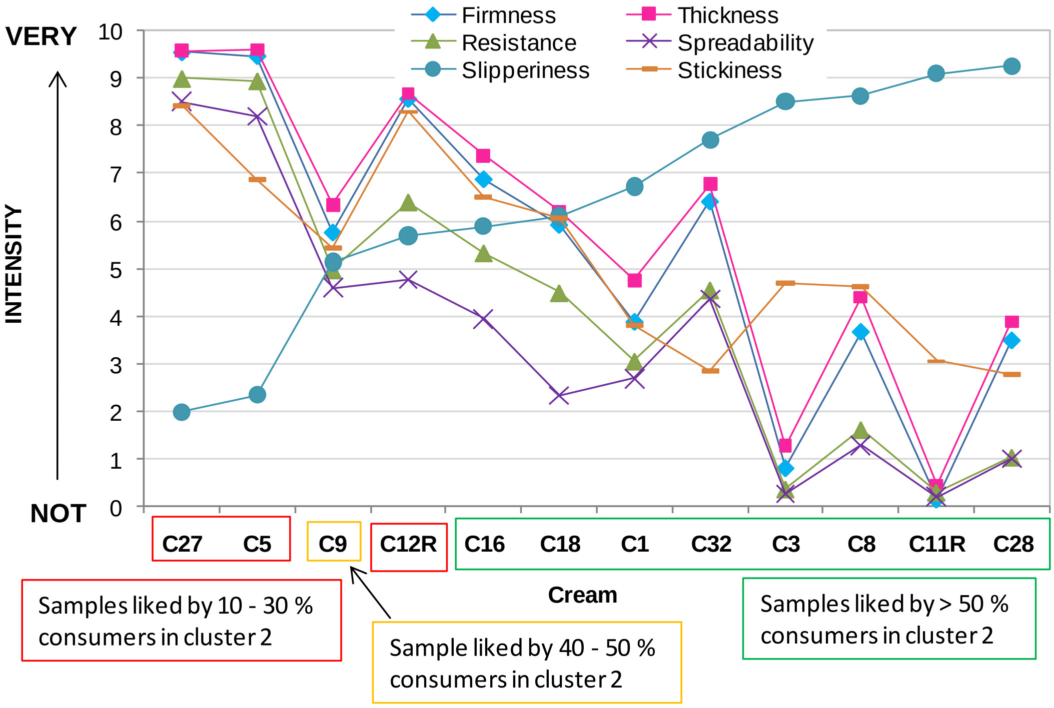


Figure 3.22: *QDA scores for the attributes on PC1 plotted in order of increasing slipperiness illustrating the effect these sensory properties have on consumer liking.*

Consumer liking behaviour: Vector models, cluster three

The direction of liking for consumers in cluster three appears to be towards the negative side of PC1 and the positive side of PC2 hence the majority of consumers are positioned in the top left hand quadrant (see Figure 3.23). Looking at the sensory scores for creams liked by 60 – 100 % of consumers in cluster 3 (creams 1, 3, 8, 11R and 28, see Figure 3.24) revealed that attributes on PC1 were playing a key role in the liking behaviour. All creams with firmness < 4, thickness < 5, resistance < 4, spreadability < 3, stickiness < 5 and slipperiness > 6.5 were liked by 60 – 100 % of consumers, whereas samples exceeding these values were liked by fewer consumers (0 – 50 %). For samples within the acceptable limits for attributes on PC1, i.e. low firmness, thickness, resistance, spreadability, stickiness (< 5) and high slipperiness scores (> 6), those with low final greasiness (≤ 5) and absorption scores between 3 and 7 were preferred (creams 1, 3, 8 and 11R). Lee et al. (2005) also found that an ease of spreading and a low degree of stickiness were desirable cream characteristics from a consumers perspective (see Chapter 1.4.2).

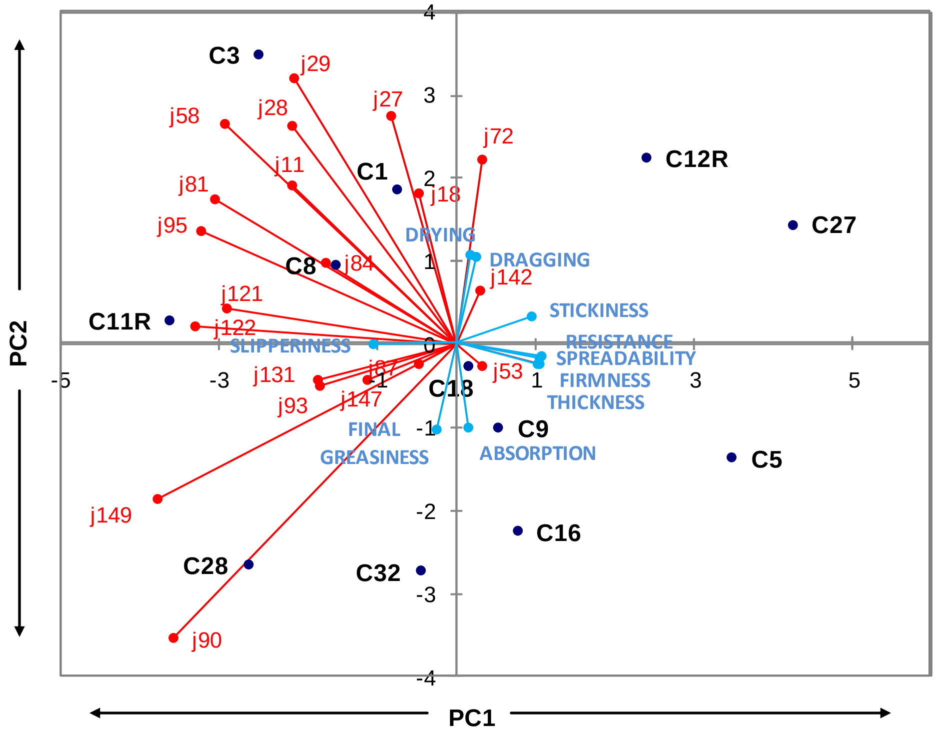


Figure 3.23: *Preference map for consumers with vector models in cluster three.*

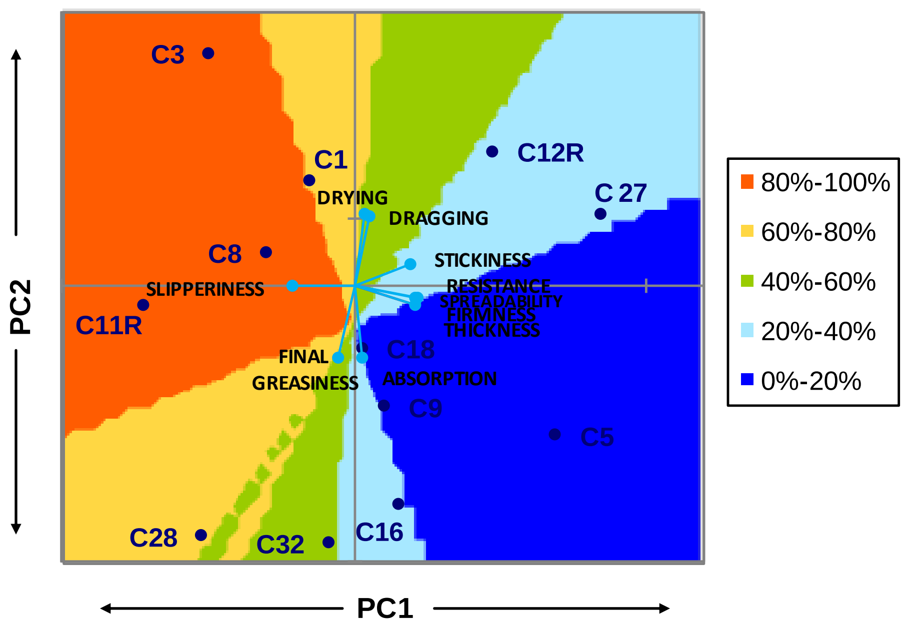


Figure 3.24: *Contour plot for consumers with vector models in cluster three.*

3.2.5.2 Non-vector models

The majority of the consumers showing ideal and saddle point models were towards the centre of the design space (see Figure 3.25). Consumers with anti-ideal models (j25 and j96) were far from the centre therefore not within the product space. In order to understand the relationship between consumer liking for the different types of model, results will be discussed separately.

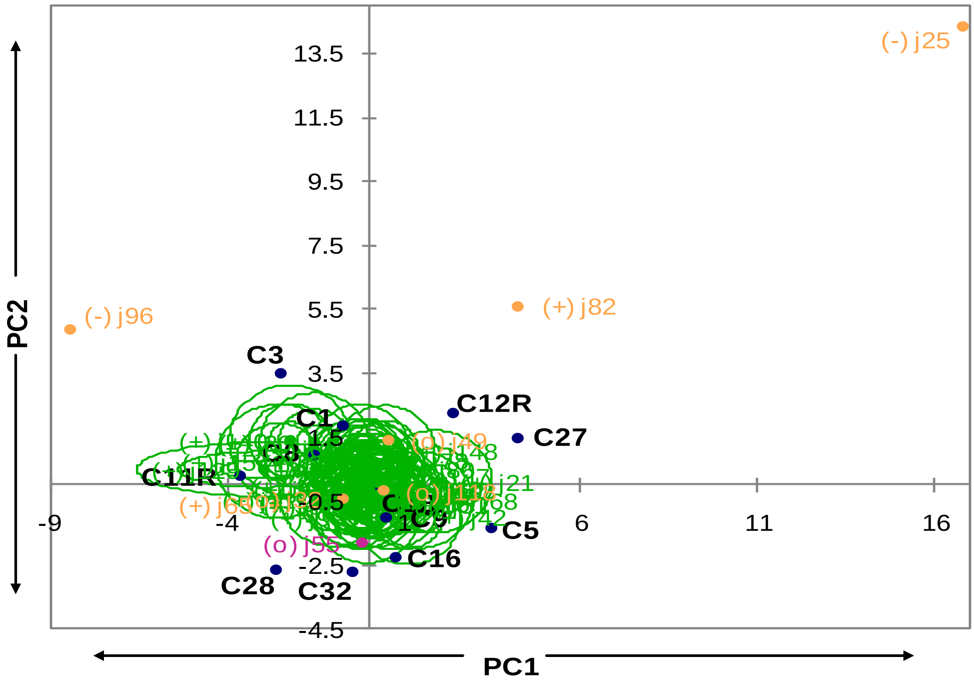


Figure 3.25: *External preference map showing ideal (+), anti-ideal (-) and saddle (o) point models. Green font and circles around the ideal points represent consumers with circular model fits. Elliptical and quadratic fits are indicated by purple and orange font respectively.*

Consumer liking behaviour: Ideal point models

The majority of ideal point models showed circular fits (91 %), the remaining percentage being quadratic (6 %) and elliptical fits (3 %). The contour plot for consumers with ideal point models is given in Figure 3.26. Results indicate that moving through the design space to more extreme samples, consumers with ideal point models like the products less. For example, creams 5 and 27 (the thickest samples), creams 11R and 3 (the thinnest samples) and cream 28 (the least drying and dragging sample with the fastest absorption and highest final greasiness scores) were liked by less than 20 % of consumers in this group (see Figure 3.26).

More than 50 % of consumers with ideal points liked creams 1, 8, 9, 16, 18 and 32. The characteristics of these more popular creams in terms of QDA scores for attributes on PC1 follow: firmness 3.5 - 7; thickness 4 - 8; resistance 1 - 6; spreadability 1 - 5; and slipperiness 5 - 9. The acceptability ranges vary for the different attributes. For firmness and thickness, it is clear that extreme samples are disliked (i.e. very firm or thick and not very firm or thick), whereas for the resistance and spreadability, liked samples are towards the lower end of the scale (1 - 6). This suggests that higher extremes for resistance or difficulty of spreading are much less acceptable than low resistance or difficulty of spreading.

Attributes on PC2 also play a role in liking behaviour of ideal point model consumers although these relationships are less dominant. Creams liked by 90 – 100 % of the ideal point model consumers had the following attribute properties: drying 1.6 - 6.3; dragging 1.6 – 5.6; absorption 5 – 7 and final greasiness 0.9 – 5. Note that the characteristics of attributes on PC1 dominated the consumer liking behaviour. For example cream 11R was within the drying, dragging, final greasiness and absorption ranges liked by 90 – 100 % consumers but it had extreme properties in terms of attributes on PC1 (see Figure 3.22) hence it was only liked by 10 – 20 % of ideal point consumers.

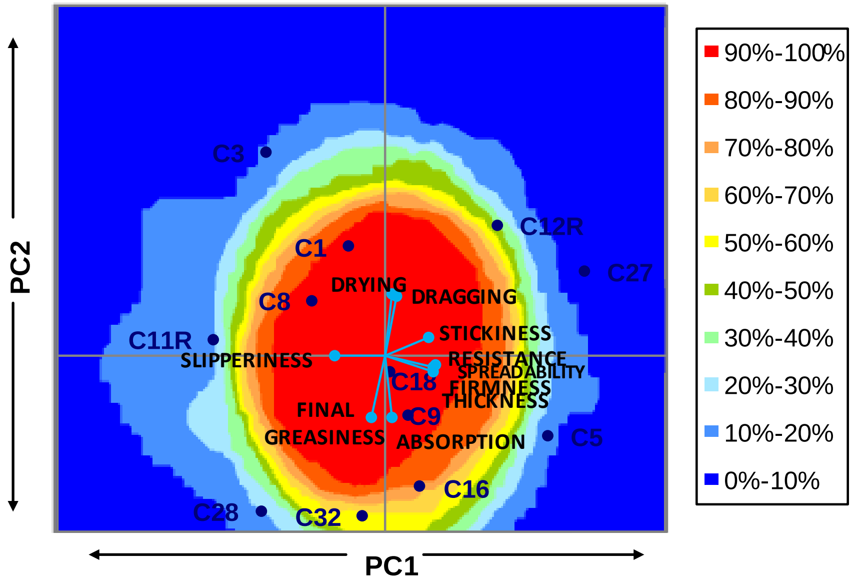


Figure 3.26: *Contour plot showing the percentage of ideal point consumers satisfied the different creams.*

Consumer liking behaviour: Anti-ideal and saddle point models

The anti-ideal and saddle point models showed much more complex relationships; there were only six consumers expressed by these models (two consumers with anti-ideal point models and four consumers with saddle point models). Consumers with saddle point models liked a mixture of sensory properties although it was clear that extreme samples for attributes towards the positive side of PC1 were disliked. Acceptable ranges for attributes on PC1 liked by these consumers follow: firmness and thickness 3 - 9; resistance 1 - 7 and spreadability 1 - 5. Results for anti-ideal models were less clear, however, results obtained for these models were outside the product space (see Figure 3.25). Therefore the amount of useful information that could be gained from analysing these types of model was limited.

3.2.6 Consumer liking in terms of satisfaction and dissatisfaction

Average DIS (dissatisfaction) and SAT (satisfaction) scores for all consumers were calculated and plotted against sensory attributes as described in Chapter 2.3.3.5. Then DIS and SAT scores for consumers showing different types of model (ideal point, vector models) were looked at separately to allow for further interpretation of these results.

Attributes on PC2 along with stickiness showed limited trends with liking behaviour of consumers for the different model types. Trends observed for the attributes firmness and thickness were in the form of inverted u-shapes for the ideal point models, saddle point models and vector models clusters one and two, thus agreeing with previous results which suggested that the majority of consumers disliked the extremely thin and thick samples. Consumers with vector models in cluster three showed minimal DIS/SAT liking trends while consumers with anti-ideal point models showed a linear liking trend (as the firmness and thickness increased, liking decreased). Figure 3.27 illustrates the inverted u relationship described above (see also Figure 2.10, Chapter 2.3.3.5 for thickness).

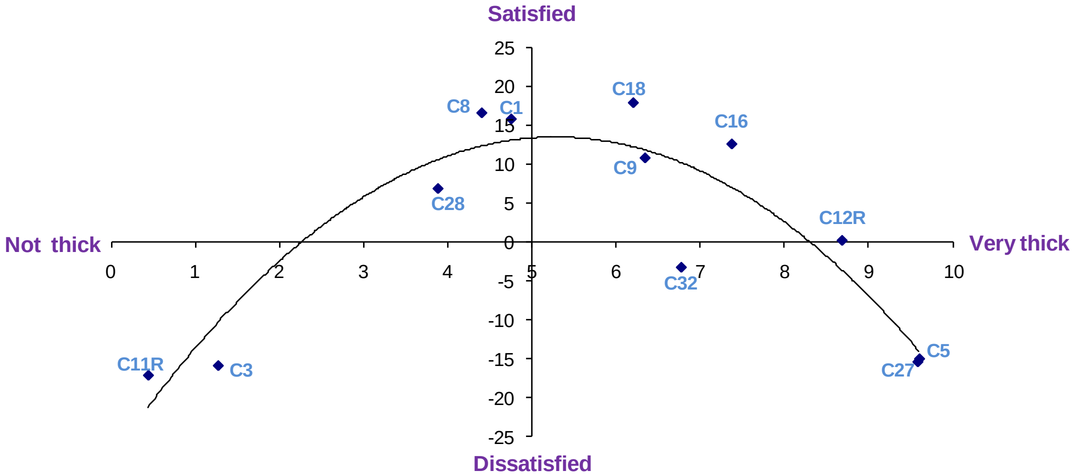


Figure 3.27: *Average dissatisfaction and satisfaction scores for consumers with ideal point models as plotted against QDA thickness data.*

The most common relationships observed for the attributes resistance and spreadability showed that increasing the resistance (or difficulty of spreading) caused a decrease in liking. However, there were exceptions to the rule. For example, creams 3 and 11R and in some cases cream 28 all had resistance and spreadability values ≤ 1 and they were generally liked less (greater dissatisfaction), therefore this satisfaction trend was also an inverted u shape although it is skewed to the left, towards the 'easy to spread' anchor (see Figure 3.28). Once again, this emphasises the fact that in general consumers did not like the extreme samples. This trend was less obvious for consumers with vector models in cluster three and consumers with saddle point models.

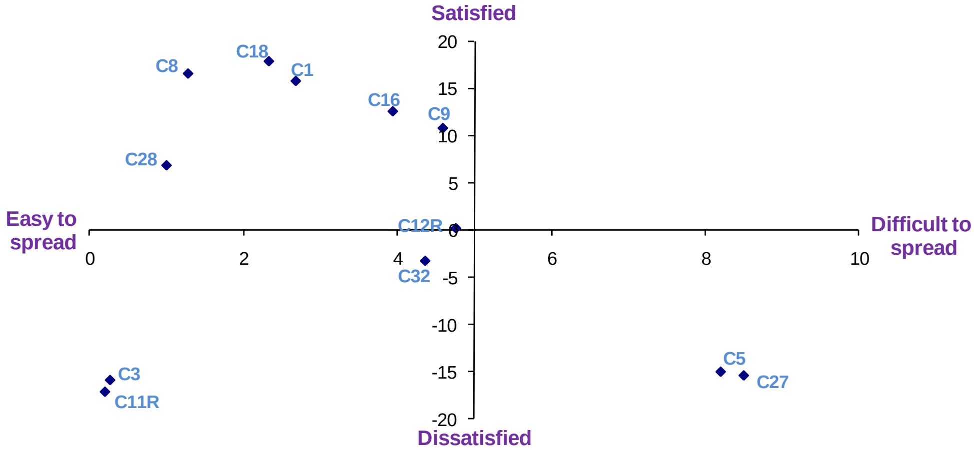


Figure 3.28: *Average dissatisfaction and satisfaction scores for consumers with ideal point models as plotted against QDA spreadability data.*

The inverted u trend observed in Figure 3.27 is commonly observed when analysing hedonic data (Booth and Conner, 1990). Theoretically, hedonic results for individual consumers should peak forming an inverted v trend ('acceptance triangle') (Booth and Conner, 1990) since individuals tend to have a preferred level for particular factors of different products. Therefore products with levels of the factor above or below the ideal would be liked less by the consumer. On the other hand, when observing trends in averaged consumer liking data, the trend has a rounded top (u shape) since different consumers have different ideals (Shepherd et al., 1989).

Berlyne (1970) developed an arousal theory that relates consumers liking of a product with arousal which is thought to be directly related to the novelty of the stimulus. If the stimulus is too complex, consumer liking decreases, alternatively if the stimulus is not complex enough (too familiar) boredom sets in and the consumer liking decreases. These factors lead to the inverted u shape (Levy et al., 2006). The optimum level of complexity liked by a consumer is thought to increase with repeated exposure to the product (Dember and Earl, 1957) hence liking is related to familiarity. As well as familiarity, Sluckin et al. (1980) report that liking is related to time, whereby liking for a new product increases with time and then slowly declines.

The majority of consumers in this study were regular hand cream users (see Chapter 3.2.1, Figure 3.11) therefore they were familiar with cream products found on the market. Consumers were informed that products in this study were ‘hand cream bases’ (see Chapter 2.3.3.4), for this reason the inverted u trends observed in this research are likely to reflect the properties of hand creams used by consumers on a regular basis. Results confirm this hypothesis as in general, samples that were disliked were extreme cream formulations i.e. products that may not be classed as regular ‘hand creams’ on the market. For example cream 11R was very thin and closer to the texture of a lotion rather than a hand cream and cream 5R was very thick, close to the texture of Sudocrem (an antiseptic healing cream, Forest Tosara Ltd., Ireland). Consumers were generally dissatisfied with these samples see Figure 3.27.

In hindsight this method of observing consumer liking in terms of satisfaction and dissatisfaction has not added any new information to the cluster analysis and external preference mapping results. The classical ‘attractive’, ‘performance’ and ‘must be’ attribute trends were not observed in this case (see Chapter 1.4.3.4). However, it was a useful technique for visualising the trends in liking behaviour for different clusters and model types. Therefore used alongside cluster analysis and preference mapping it can provide a more efficient way of looking at the liking trends than looking at raw data.

These results including those from AHC and external preference mapping have confirmed that in general consumers dislike extreme cream samples. If this study were to be carried out again it would be worth replacing some of the extreme samples with creams closer to the middle of the design space (or using a larger sample set containing more samples closer to the middle of the design space) to get a better idea of consumer liking behaviour in relation to sensory properties found in products on the market.

3.2.7 Summary

Overall it was found that creams towards the centre of the PCA plot (design space) were liked more by the majority of the consumers i.e. extreme cream samples were generally disliked.

Key factors that affect consumer liking appear to be: ***1) Firmness and thickness of the sample*** - extreme samples in terms of firmness and thickness were generally disliked i.e. creams that were really firm and thick or not at all firm or thick. Desirable samples therefore had some form of structure but not too much, this is related to the second finding; ***2) How easy it was to apply the cream to the skin*** - samples with high resistance and high difficulty of spreading were generally disliked suggesting consumers prefer samples that are easy to apply to the skin (high resistance and difficulty of spreading relate to thicker samples which are more difficult to spread on the skin). Samples with extremely low resistance and difficulty of spreading were also disliked, such samples may also be difficult to apply to the skin as low resistance suggests a runny or slippery sample which could be difficult to transfer from the container to the skin; ***3) How well the cream provided moisture to the skin*** - samples that were drying or dragging in nature were generally disliked by consumers, likewise samples with slower absorption rate and higher final greasiness were preferred suggesting consumers were judging liking in terms of how well the cream functions in providing grease to the skin. If the absorption is too quick or the final greasiness of the skin after absorption is low it could suggest limited performance of the cream as a moisturiser.

3.3 RHEOLOGICAL PROPERTIES

Rheological properties were measured according to the oscillation amplitude sweep, frequency sweep and steady shear protocols described in Chapter 2.4.1. Various parameters were analysed from the different test methods and in all cases both average and standard deviations (SD) were calculated. The oscillatory and steady shear measurements were repeated on the 12 consumer study creams (that were produced as a fresh batch) with minor alterations to the measurement protocols (see Chapters 2.4.1.1.2 and 2.4.1.2.2).

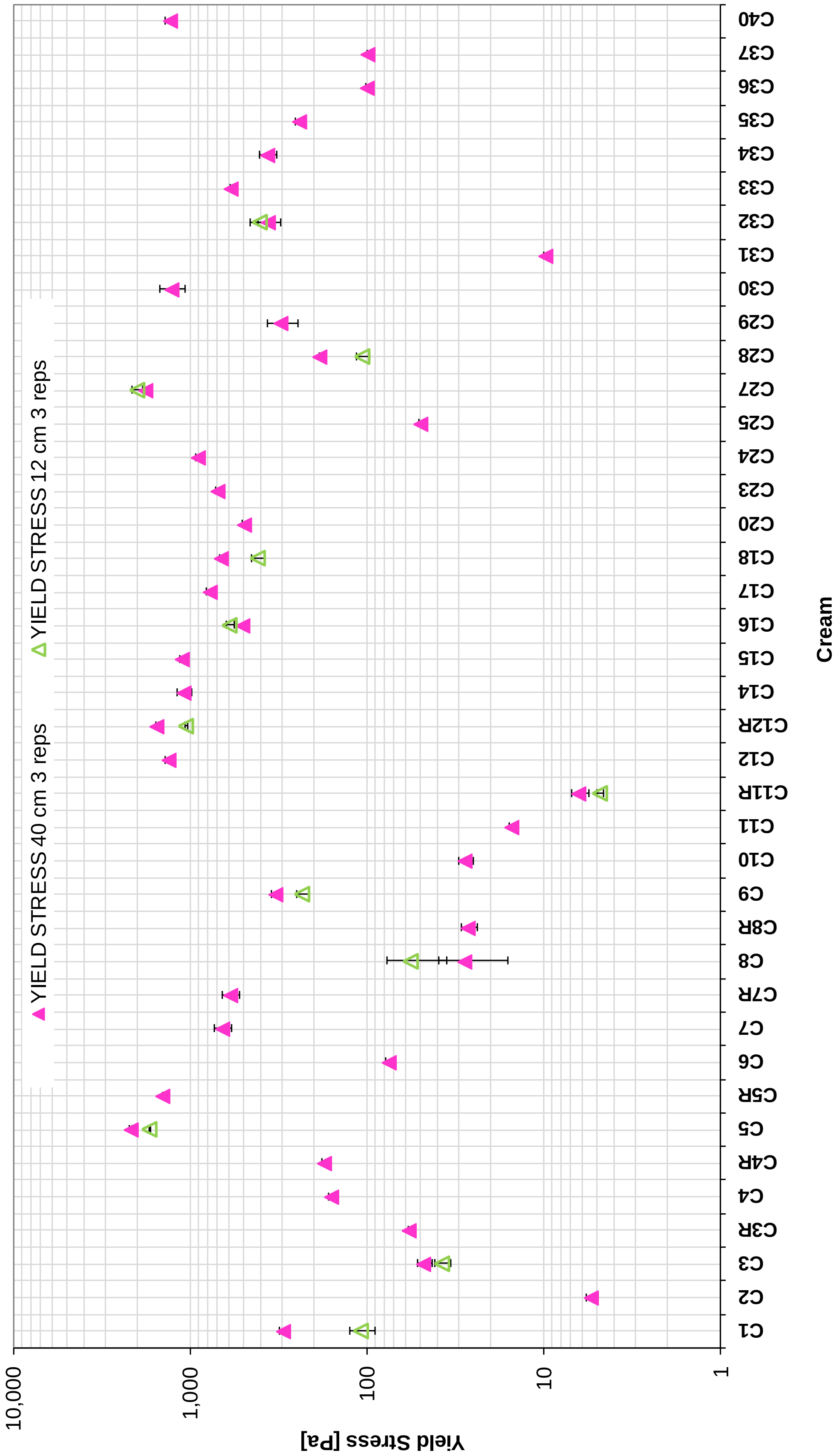
Comparing the results obtained from measuring the 40 creams with the 12 consumer study creams showed some discrepancies. The difference between results was larger for some samples than others suggesting inhomogeneity in the samples. However, it should be noted that rheological measurements were carried out on the 40 creams approximately seven months after cream manufacture over a period of four months, while the time frame for measuring the 12 creams was within three weeks, measured one month after manufacture. These differences in time frame between manufacture and measurement are likely to be responsible for differences in data due to the cream structure changing over time. Note that the greater reproducibility of results obtained for sensory data (see Chapter 3.2.2) are due to the sensory measurements being taken closer to skin cream manufacture. In this case the 40 creams were measured two months after manufacture over a period of four months while the 12 creams were measured within three weeks, one month after manufacture.

3.3.1 Oscillation amplitude sweep

Typical results obtained from oscillation amplitude sweep measurements were shown in Figure 2.11 (Chapter 2.4.1.1.1). At low strains, the G′ values were higher than the G″ values for all creams indicating elastic behaviour of the samples. Upon increase in strain, G′ values started decreasing and the LVD was overcome, this occurred at γ ~10 – 100 % for the majority of samples. On further increase of strain, increasing dominance of viscous flow effects was revealed by a comparatively steeper decrease of G′ to G″ leading to cross over.

Yield stress results as calculated following the Walls et al. (2003) method (see Chapter 2.4.1.1.1, Figure 2.12) confirmed that a broad spectrum of textures were produced by the original experimental design, see Figure 3.29 and Tables A8.1 and A11.1, Appendices VIII and XI. Results for replicate samples (as indicated by R) were similar indicating good batch-to-batch consistency of skin cream production. Repeat measurements on the same sample showed good repeatability. The coefficient of variation, CV, (CV = (SD/average)*100 %) was < 20 % for all samples except cream 8 (43 %). It is possible that this formulation was unstable as results obtained when measuring the consumer study creams also showed high error for cream 8 (38 %). This hypothesis is supported by the fact that the first measurement carried out on cream 8 resulted in much higher G′ values than were found in repeat measurements which were carried out over a time frame of 4 months (40 cream 3 replicates) and 3 weeks (12 creams 3 replicates) respectively. This suggests the sample structure is breaking down with time. Interestingly, the error for cream 8R, the replicate sample was low (10.5 %). The rheological data for this sample also showed a decrease in G′ between the first 2 measurements but this sample increased in viscosity slightly with further measurements (hence the lower coefficient of variation) also confirming a potential stability problem and pointing towards sample inhomogeneity.

Figure 3.29: *Average yield stress values for model skin creams as obtained from triplicate oscillation amplitude sweep measurements. Error bars giving the standard deviation (SD) ranges are included.*



The lowest average yield stress recorded for the creams involved in this study was 5 Pa (C2 and C11R) and the highest was 2174 Pa (C5). This difference in yield stress of approximately 2000 Pa emphasises the extremes involved in this study (very thin to very thick creams). Brummer and Hammer (1997) state that the onset of flow for creams is generally above a critical shear stress of 10 Pa, whereas lotions begin to flow below 10 Pa. In this research, creams 2, 11 and 31 all have yield stresses < 10 Pa indicating their rheological behaviour is closer to a lotion than a cream.

Cream 5 (highest yield stress) contained the highest levels of all variable ingredients where oil type was silicone oil and thickener was carbopol (see Table 2.2, Chapter 2.2.1). The high oil, thickener and SA levels would have provided body to the sample resulting in a thick cream (Eccleston, 1986; Epstein, 2009). This combination of ingredients would have allowed for sufficient neutralisation of the SA by the TEA to create a well-dispersed system with a strong internal structure resulting in the high yield stress (see Chapter 1.5.2.2). Cream 40 also contained the highest levels of all variable ingredients although in this case the oil type was mineral oil and the thickener was veegum. Analysis of ingredient effects on formulation properties showed that oil type did not affect the firmness or thickness of the sample. Veegum however imparts less structure to the sample than an equivalent formulation containing Carbopol (Braun, 1991), therefore, cream 40 had slightly lower values than cream 5 for yield stress (see Chapter 2.2.1).

Cream 2 (lowest yield stress) on the other hand contained the lowest levels of all variable ingredients except for TEA where the highest level was present. Cream 11R contained a medium oil level (20 %), the highest level of TEA (5 %), the lowest level of SA (5 %) and no thickener. All samples containing ≤ 20 % oil, 5 % SA and veegum if thickener was present (C2, C6, C8, C8R, C10, C11, C11R, C31 and C37) had yield stresses < 100 Pa. Of these samples, those containing 5 % TEA (C2, C11, C11R and C31) had very low yield stresses (< 20 Pa). The low levels of SA, oil and thickener (structuring agents) are responsible for the yield stresses < 100 Pa while the TEA level appears to affect how low the yield stress will be.

Creams 6 and 10 contained the same levels of ingredients as cream 2 except for TEA where the lowest level (0.5 %) and the medium level (2.75 %) were present respectively. It is interesting that the yield stresses of these low SA cream formulations (C2 = 5 Pa; C6 = 75 Pa; C10 = 28 Pa) appear to be related to the TEA level where a high level results in a relatively low yield stress (C2) and vice versa (C6). This suggests that the overall ratio of SA to TEA (and therefore the level of neutralisation) affects the structure of the cream.

Neutralisation levels were calculated for all model skin cream formulations ((Mol TEA/Mol SA)*100 %). Complete neutralisation of SA by TEA to form triethanolamine strearate occurred in all formulations containing medium to high levels of TEA (2.75 % or 5 %) and low levels of SA (5 %) (C2, C4, C4R, C10, C11, C11R, C16, C25, C31, C37). The high levels of neutralisation meant that all the SA and TEA had formed triethanolamine stearate (the soap which acts as an emulsifier) within the formulation. This will have prevented the SA providing any structure to the sample (see Chapter 2.2.1) leading to the comparatively lower yield stresses observed in these samples.

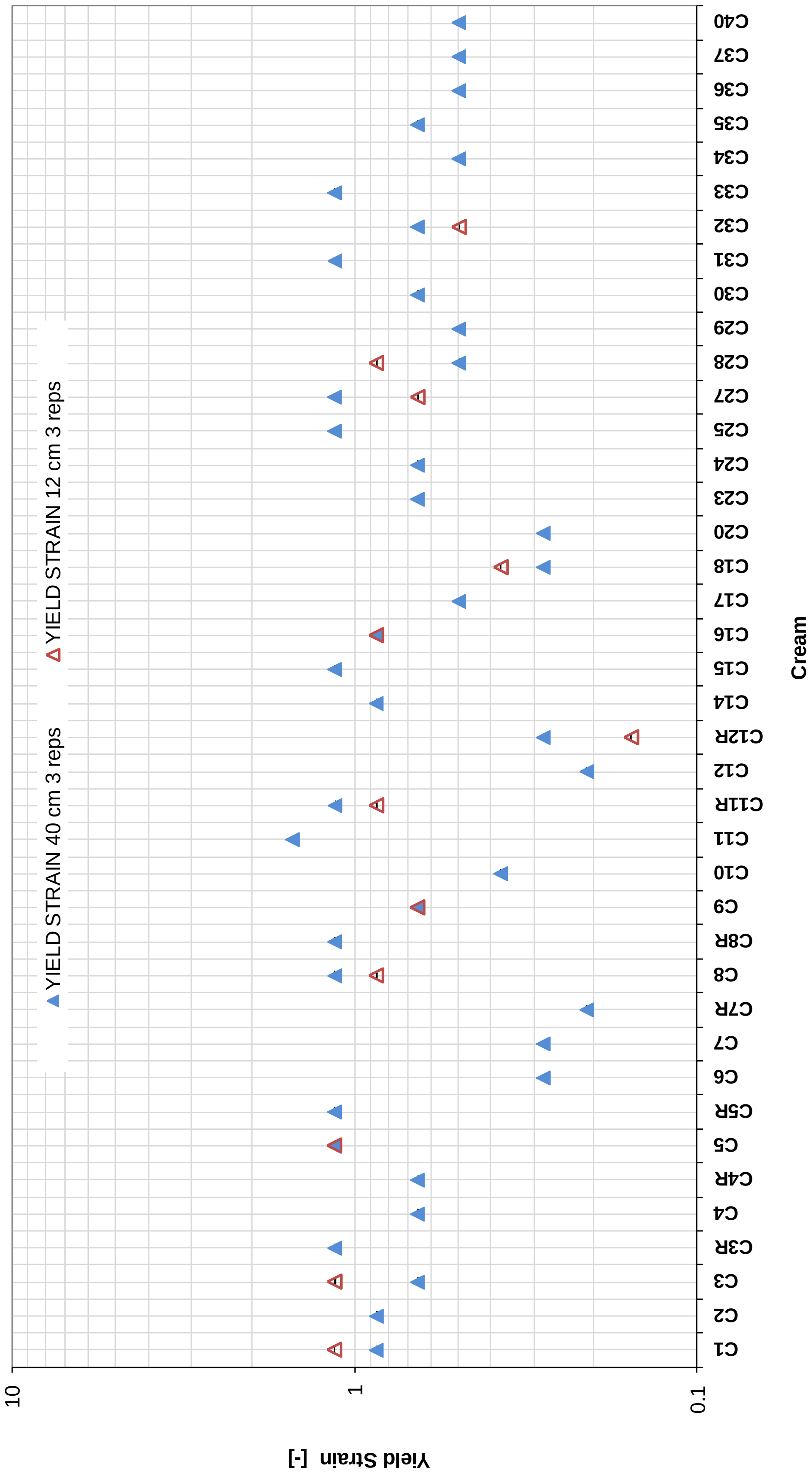
Of the samples in which complete neutralisation occurred, those containing Carbopol had yield stresses > 90 Pa (C4, C4R, C16 and C37) while the others (containing no Carbopol) had yield stresses < 50 Pa. This suggests that as well as the SA:TEA ratio, thickener type and level also play a role in the resulting yield stress of a sample as becomes clear when comparing the results for creams 25, 28 and 35. These 3 samples all contain 40 % oil, 5 % SA, 0 % thickener and differing levels of TEA (C25 contains 5 % TEA while C28 and C35 contain 0.5 %). The lower level of TEA in creams 28 and 35 means that these samples have undergone 19 % neutralisation which has resulted in a considerably higher yield stress (C28 = 185 Pa; C35 = 241 Pa) than found for cream 25 (49 Pa). Other samples in which 19 %

neutralisation has occurred include creams 6, 8, 8R, 33 and 36, of these samples, those containing oil or Carbopol had yield stresses > 90 Pa while those containing no oil or Carbopol had yield stresses < 80 Pa. This demonstrates that as well as the neutralisation level, the oil and Carbopol content play a role in the resulting yield stress.

Typical neutralisation levels for skin creams range between 20 and 40 % (Telford, 2007) but this does depend on the rest of the formulation, for example the oil and thickener levels as discussed above. Creams in this study with neutralisation between 25 and 50 % all had yield stresses > 400 Pa. The model creams used in this study included extreme formulations to enable as broad an understanding as possible of sensory and rheological properties of different skin cream formulations. Therefore the standard levels of neutralisation were not applied for all formulations.

Yield strain values obtained for the 40 skin cream samples and the 12 creams used in the consumer study are given in Figure 3.30. See also Tables A8.1 and A11.1 in Appendices VIII and XI respectively. These values may provide an indication of the stretchiness of the samples (Hopkinson and Williams, 2007), see Chapter 2.4.1.1.1. A higher yield strain (strain at which the yield stress was taken) suggests that sample is stretchier because more force must be applied before the sample flows. Creams 3R, 5, 5R, 8, 8R, 11, 11R, 15, 25, 27, 31 and 33 were the ‘stretchiest’ samples. Creams 12 and 7R were the least stretchy.

Figure 3.30: *Average yield strain values for model skin creams as obtained from triplicate oscillation amplitude sweep measurements. Error bars giving the SD ranges are included.*



Thickeners enhance stability, and add body to skin cream samples (Epstein, 2009). Carbopol is thought to impart draggy feel when used at higher levels (Epstein, 2009). Veegum on the other hand is reported to improve the spreadability of products (Vanderbilt, 2004). Looking at QDA scores for equivalent formulations where only thickener type differs: C1 and C30; C7 and C15; C8 and C33; C18 and C24; C20 and C27, (see Table 2.2, Chapter 2.2.1), revealed that samples containing Veegum (C1, C7, C8, C18 and C20) rather than Carbopol were indeed easier to spread. However, no correlation between 'dragginess' and Carbopol content was found. For these equivalent formulations in general, samples with higher yield strains (C5, C15, C24, C27) were more difficult to spread (see QDA scores Table 3.1, Chapter 3.1.1) indicating that stretchy samples are more difficult to spread.

Yield strain values were independent of the yield stress, for example, C12 and C5R both had similar and high yield stresses but C5R was much stretchier (higher yield strain), likewise C8 and C10 both had low yield stresses but C8 was stretchier. Differences in yield strain values for samples of equivalent yield stress provide an indication of a samples structural strength. The greater yield strain value of C5R in comparison to C12 is likely to be related to the presence of oil (40 %) and thickener (1 %) which were absent in C12. The presence of oil means that the soap formed during neutralisation of SA by TEA can emulsify the oil creating a well dispersed system with a stronger internal structure than C12. The Carbopol would also enhance the structural strength (Epstein, 2009) leading to the stretchier characteristics of C5R. The absence of oil meant that C12 contained mainly water (74 %) and was therefore relatively dilute. This means weaker van Der Waals attractive forces and therefore an overall weaker microstructure (Moulai Mostefa et al., 2006), hence the lower yield strain.

In the case of C8 and C10, the higher yield strain observed in C8 is likely to be due to the neutralisation levels. In C10, the SA has been completely neutralised so the SA cannot add further body and structure to the sample. In the case of C8, only

19 % of the SA has been neutralised allowing the free SA to add body and further structural stability. C8 also contains Veegum which enhances product stability (Vanderbilt, 2004) hence the comparatively larger yield strain value.

Samples with high yield stress yet low yield strains are beneficial from a consumer's perspective, as they will remain structured when transferring them from their container onto the skin, then during application they will break down quickly allowing for easier spreading. It is thought that easier spreading may be related to faster absorption into the skin since a sample must spread well on the skin surface before absorption can occur (Adeyeye et al., 2002). In this study samples with high yield stress and low yield strain include creams 7, 7R, 12, 12R, 18 and 20. These samples have low spreadability scores (< 5) indicating they are easy to spread and medium to high absorption scores (5 – 8) indicating medium to fast absorption. This confirms the hypothesis reported by Adeyeye (2002). However, creams 7 and 7R were exceptions with absorption scores of 2.5 – 3.6 indicating slow absorption time. The consumer study results confirm that samples with spreadability scores < 5 were preferred by consumers (see Chapter 3.2) although the liking trends regarding absorption were more complex. In general consumers preferred samples that absorbed at a slower rate, however, this depended on other attribute properties (see Chapter 3.2.5.1 and 3.2.5.2).

Values for G′, G″ and η* at the three selected strains of 0.1 %, 1 % and 100 % were also extracted from the measurement data. In accordance with the yield stress, the magnitude of the values varied over several decades. Figure 3.31 shows the G′ data. The standard deviations for repeated measurements on the same sample were generally small, however, creams 8 and 30 were exceptions with coefficient of variation values of 44 – 83 % for the lower strains (0.1 – 1 % strain, see Figure 3.31 and Tables A8.2, A8.3, A11.2 and A11.3 in Appendices VIII and XI). Cream 30 had tiny lumps throughout rather like an exfoliating product, which may have affected sampling and therefore the error between repeated measurements. Suggestions for differences in repeated measurements for cream 8 were discussed at the start of Chapter 3.3.1.

At the lower strains (0.1 – 1 %) the majority of the samples were within the LVD, therefore the values obtained were very similar (see Figure 3.31). For this reason only G′ at 1 % strain will be discussed further as replicate data for samples obtained at 1 % strain had slightly lower error than those at 0.1 % strain. As the strain reached 100 %, a large proportion of the samples had undergone considerable deformation and the values for G″ had become greater than the values for G′.

At low strains, complex viscosity results were similar to G′ values. The relationship between complex modulus, G*, and complex viscosity, η*, is expressed by

$$|G^*| = \omega|\eta^*| \tag{3.1}$$

which in terms of G′ and G″ is

$$\sqrt{(G')^2 + (G'')^2} = \omega|\eta^*| \tag{3.2}.$$

Complex viscosity (η*) results were similar to G′ values at low strains (within the LVD) due to the dominance of elastic (G′) behaviour which means that G″ has little effect on the complex viscosity. Also, measurements were taken at 1 rad.s^{-1} therefore

$$|G'| \approx |\eta^*| \tag{3.3}$$

which is the reason for similar complex viscosity and G′ values at low strain. Summary tables containing average values and coefficient of variation values for the G′, G″, and η* at 0.1 %, 1 % and 100 % strain are given in Appendix VIII and Appendix XI, Tables A8.2 – A8.4 and A11.2 – A11.4.

Values for tanδ (G″/G′) provide a measure of the ratio of energy lost to energy stored in a cycle of deformation (Adeyeye et al., 2002). Figure 3.32 shows tanδ values obtained for the 40 model skin creams at both 1 % and 100 % strain. At

1 % strain all tanδ values were below 1 which indicates that elastic behaviour is dominating and the samples are in the gel state (Mezger, 2006) i.e. they have not yet undergone complete irrecoverable deformation (see Chapter 1.5.1). At 100 % strain, some samples had tanδ values greater than 1 indicating they have been deformed and are now in the liquid state where viscous behaviour dominates. Other samples were still in the gel state at 100 % strain.

Comparing the yield stress of a sample with its tanδ values at different strains provides useful information about how a sample will behave when applied to the skin. It is not necessarily thicker samples (i.e. those with higher yield stresses) that have tanδ < 1 at 100 % strain, some thick samples have tanδ > 1 at 100 % strain (e.g. cream 12R) indicating clear dominance of viscous behaviour. Creams 5R and 12R have similar high yield stresses (1438 Pa and 1554 Pa respectively) but cream 5R has a tanδ < 1 at 100 % strain while for cream 12R tanδ is > 1. This indicates that cream 12R has a weaker microstructure (see Chapter 1.5.2.3), suggesting it is easier to spread than cream 5R. Sensory QDA results confirm this where cream 5R was difficult to spread (QDA score = 8.2) while cream 12R was easy to spread (QDA score = 4.4). Likewise creams 4 and 28 have similar yield stresses (158 Pa and 185 Pa respectively) yet the tanδ value for cream 4 is < 1 (QDA spreadability score = 4.2) while for cream 28 tanδ > 1 (QDA spreadability score = 1.3). Results confirm the theory that for samples with similar yield stresses yet different tanδ results, those with tanδ > 1 will be easier to spread than those with tanδ < 1. Summary tables of the oscillation amplitude sweep tanδ results are given in Appendix VIII, Table A8.5 and Appendix XI, Table A11.5.

Figure 3.31: *Average G' (γ = 0.1 %, 1 % & 100 %, ω = 1 rad.s^{-1}) for model skin creams as obtained from triplicate oscillation amplitude sweep measurements. Error bars giving the SD ranges are included.*

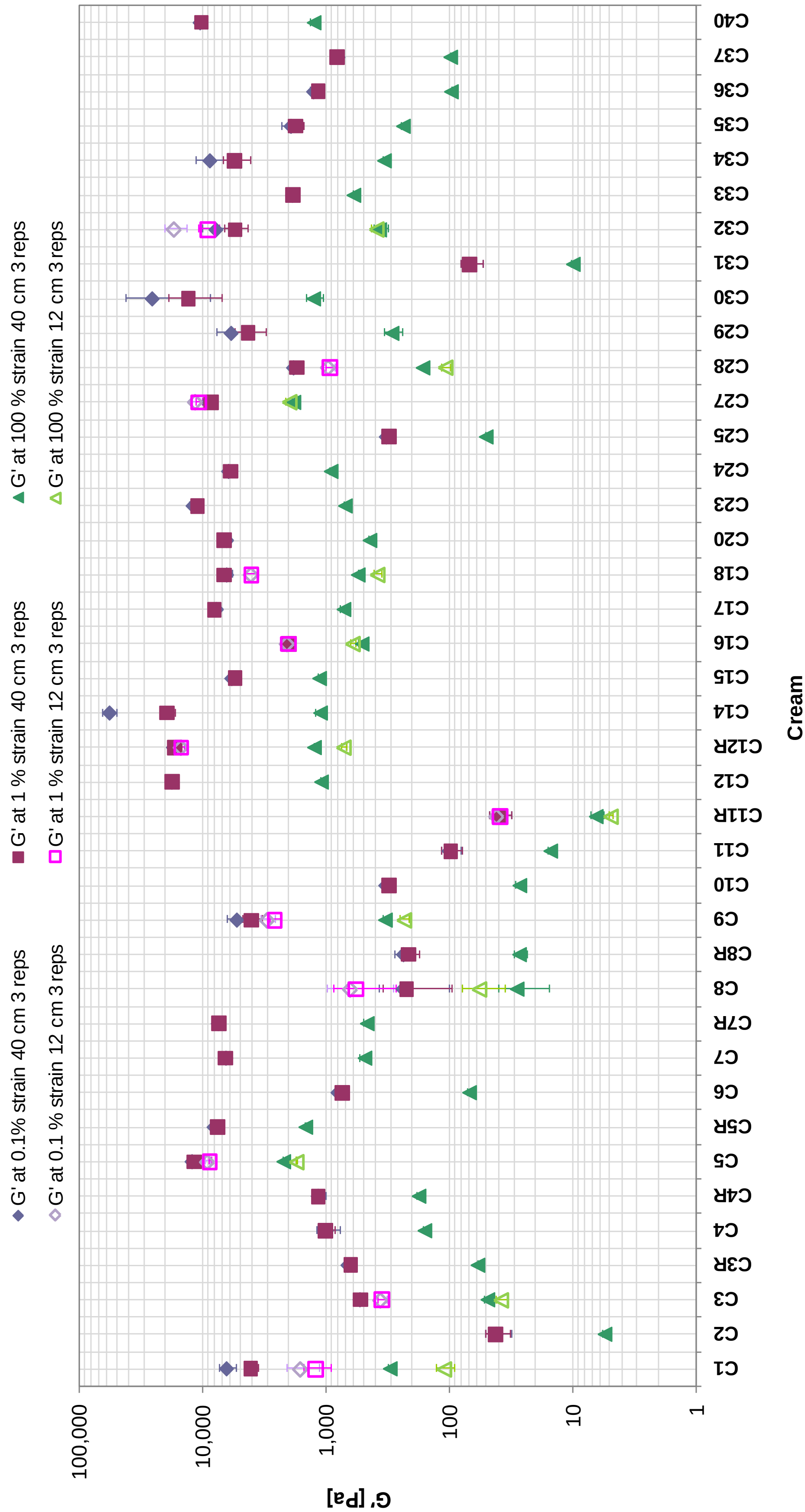
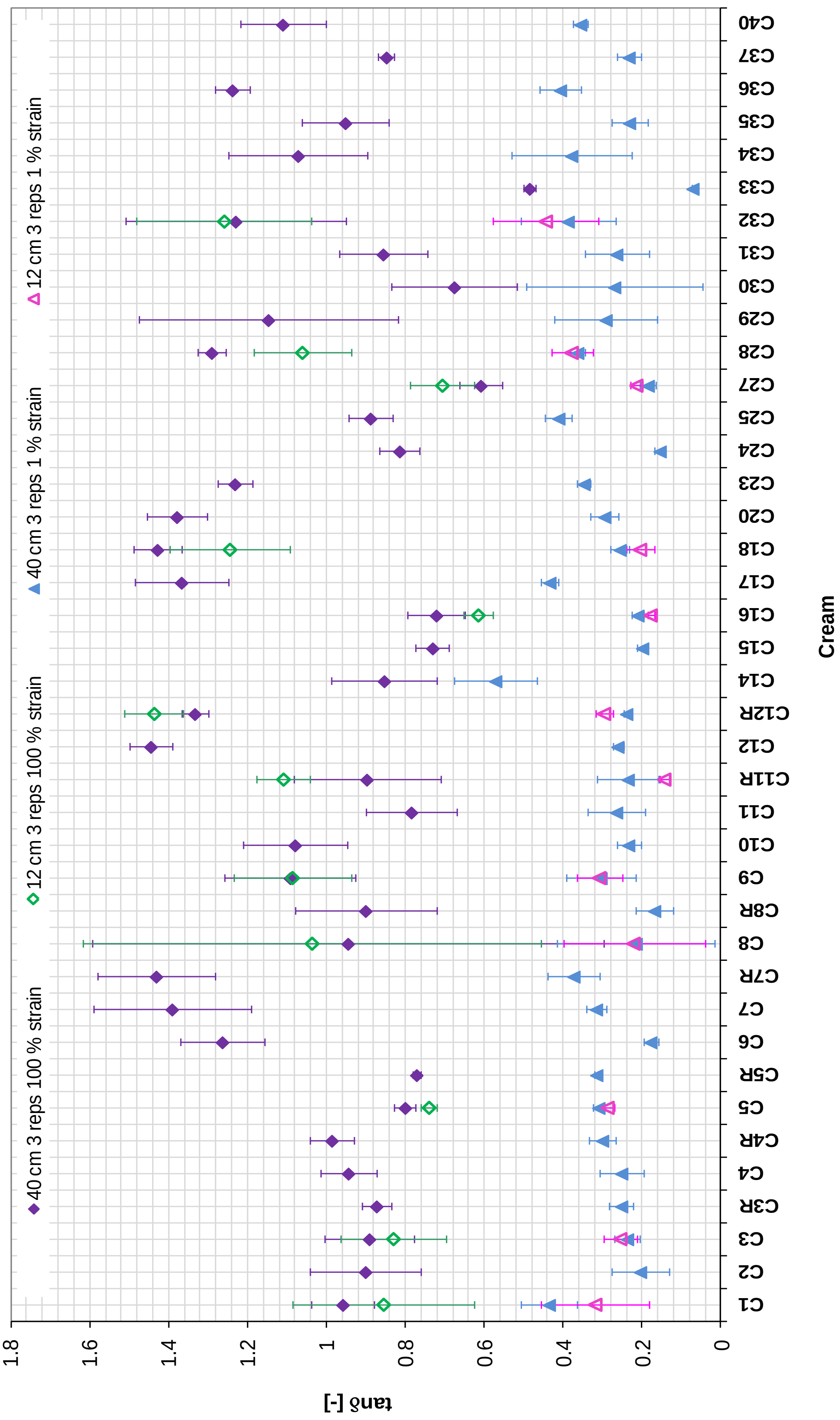


Figure 3.32: *Values for tanδ (ω = 1 rad.s^{-1}) for model skin creams as obtained from triplicate oscillation amplitude sweep measurements at 1 % and 100 % strain. Error bars giving the SD ranges are included.*



3.3.2 Oscillation frequency sweep

Parameters for the frequency sweep were selected based on preliminary tests that indicated 1 % strain should be in the LVD. However, it was revealed that the LVD in the case of cream 14 extended only to 0.2 % strain. In order to check data quality, the G′ at 1 % strain from amplitude sweep measurements and the G′ at 1 rad.s^{-1} from frequency sweep measurements were compared. Results showed values were within 20 % of each other (exceptions were creams 8R and 29 with ~25 % difference).

Figure 3.33 gives an example of frequency sweep results for three cream samples, creams 11, 4 and 12R ranging from thin to thick. The line fits for the G′ and G″ data were calculated as described in Chapter 2.4.1.1.2. For some samples the fit was poor, especially for G″ (for example, for cream 6, an R^2 of 0.1 was found). Values with poor fit were not included in the predictive modelling.

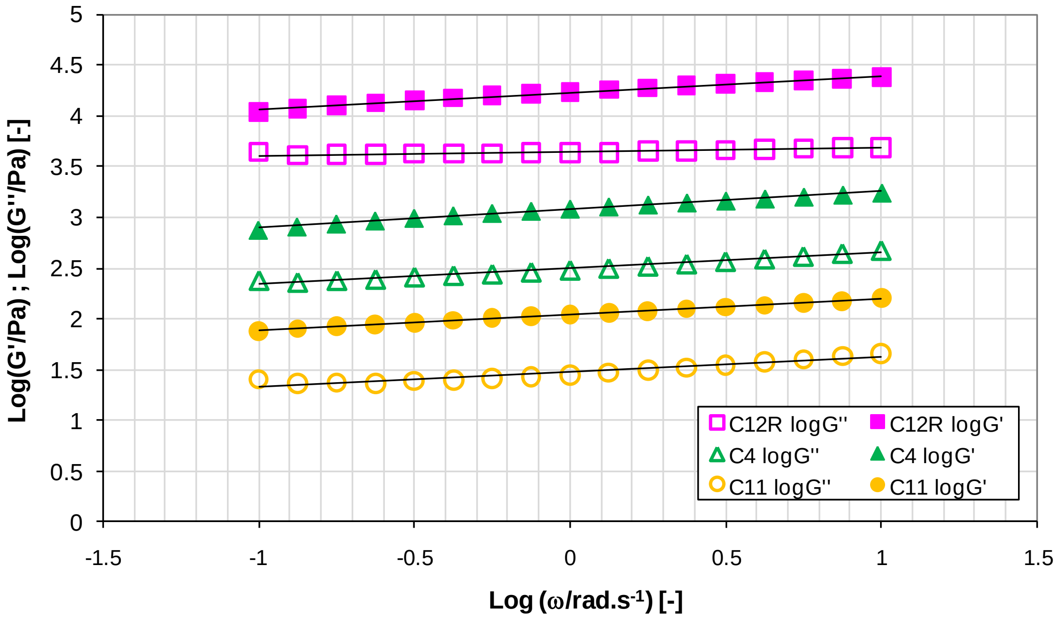
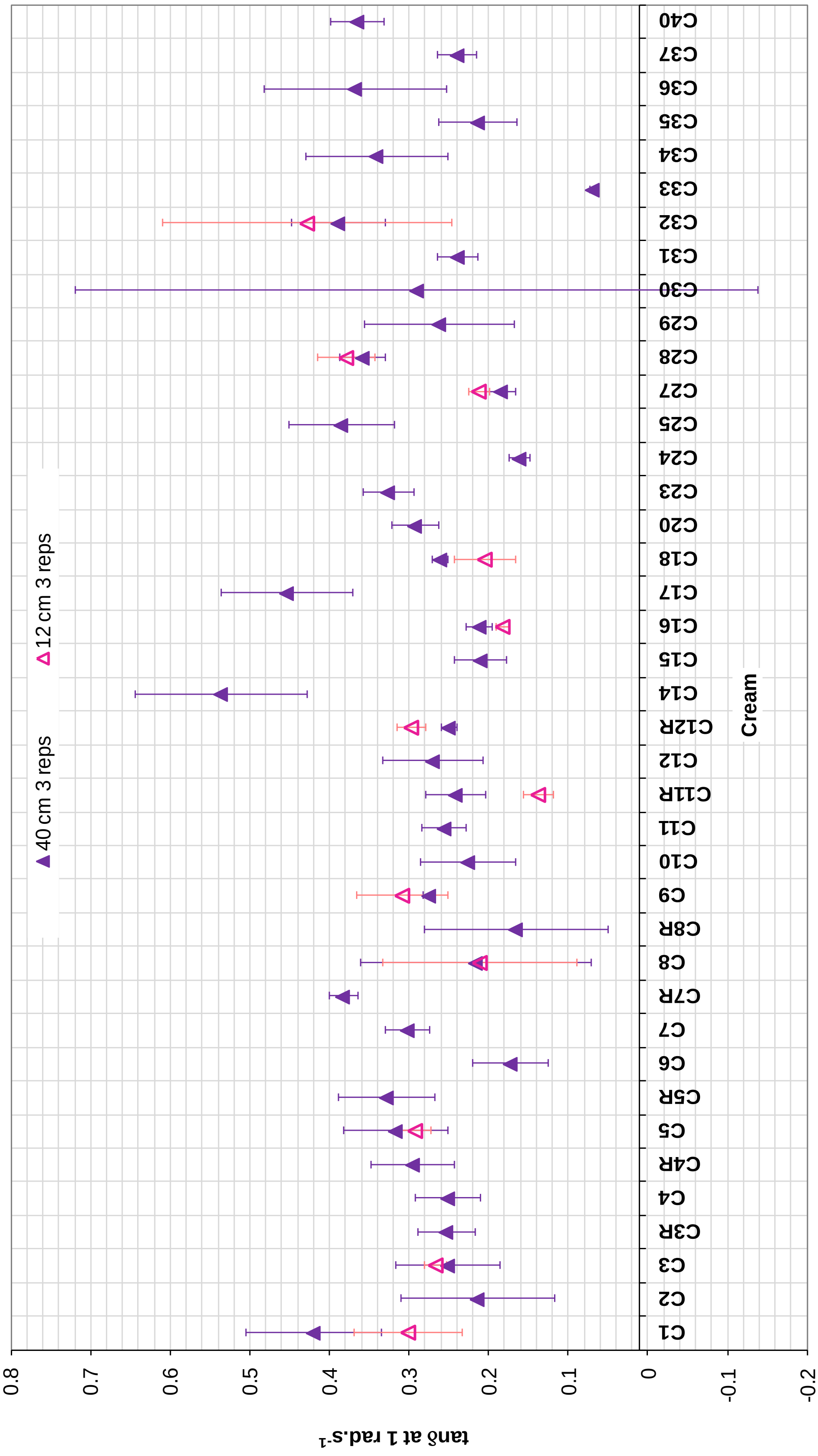


Figure 3.33: *Average frequency sweep results for three model skin cream samples, creams 11, 4 and 12R illustrating thin to thick behaviour respectively.*

The slopes of the logG′-logω and logG″-logω lines are characteristic for different types of material behaviour. A slope of approximately zero for G′ indicates gel character (Mezger, 2006). Results showed that the slopes of the logG′ versus logω plots ranged between 0.02 and 0.3, indicating 'close to gel-like' behaviour for all samples. For stable emulsions the G′, G″ lines should be parallel (Brummer, 2006), which was observed for most of the samples. Tables of average results for each cream sample are given in Appendices IX and XI, Tables A9.1 – A9.2 and A11.6 – A11.7.

For all 40 model skin cream samples the G′ values were higher than the G″ values with tanδ values at 1 rad.s^{-1} ranging from 0.07 to 0.54, see Figure 3.34 and Appendices IX and XI, Tables A9.2 and A 11.7. In hindsight, the frequency sweep results did not really add any further information to the study than that obtained through oscillation amplitude sweep measurements. Therefore if future work investigating skin creams was to be carried out, the amplitude sweep would be recommended as it provides more information about material behaviour under different conditions.

Figure 3.34: *Values for tanδ at 1 rad.s^{-1} for model skin cream samples as obtained from triplicate frequency sweep measurements. Error bars indicating SD of the replicates are given.*



3.3.3 Low shear measurements

An example of steady shear results for the lower end of the flow curve as obtained for three samples is given in Figure 3.35. This region of the flow curve was examined in stress controlled mode, see Chapter 2.4.1.2.1 for protocol. The majority of samples showed a zero shear viscosity plateau and at higher stresses shear thinning behaviour was observed as illustrated by the decrease in viscosity. For some samples the decrease in η was rapid whereas for others a less steep slope indicated a gentler transition from reversible to irreversible deformation.

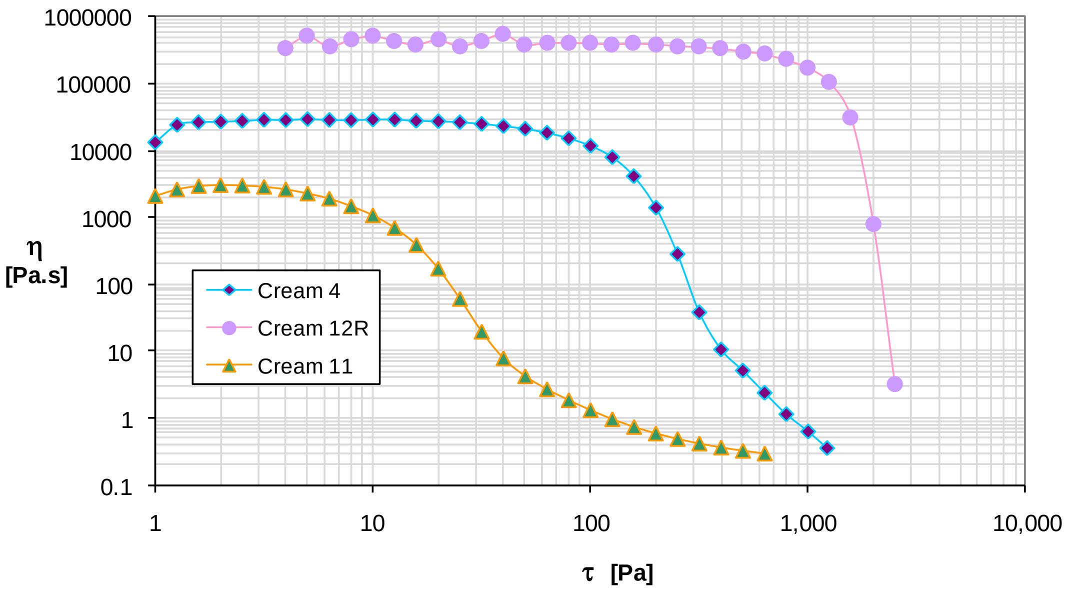
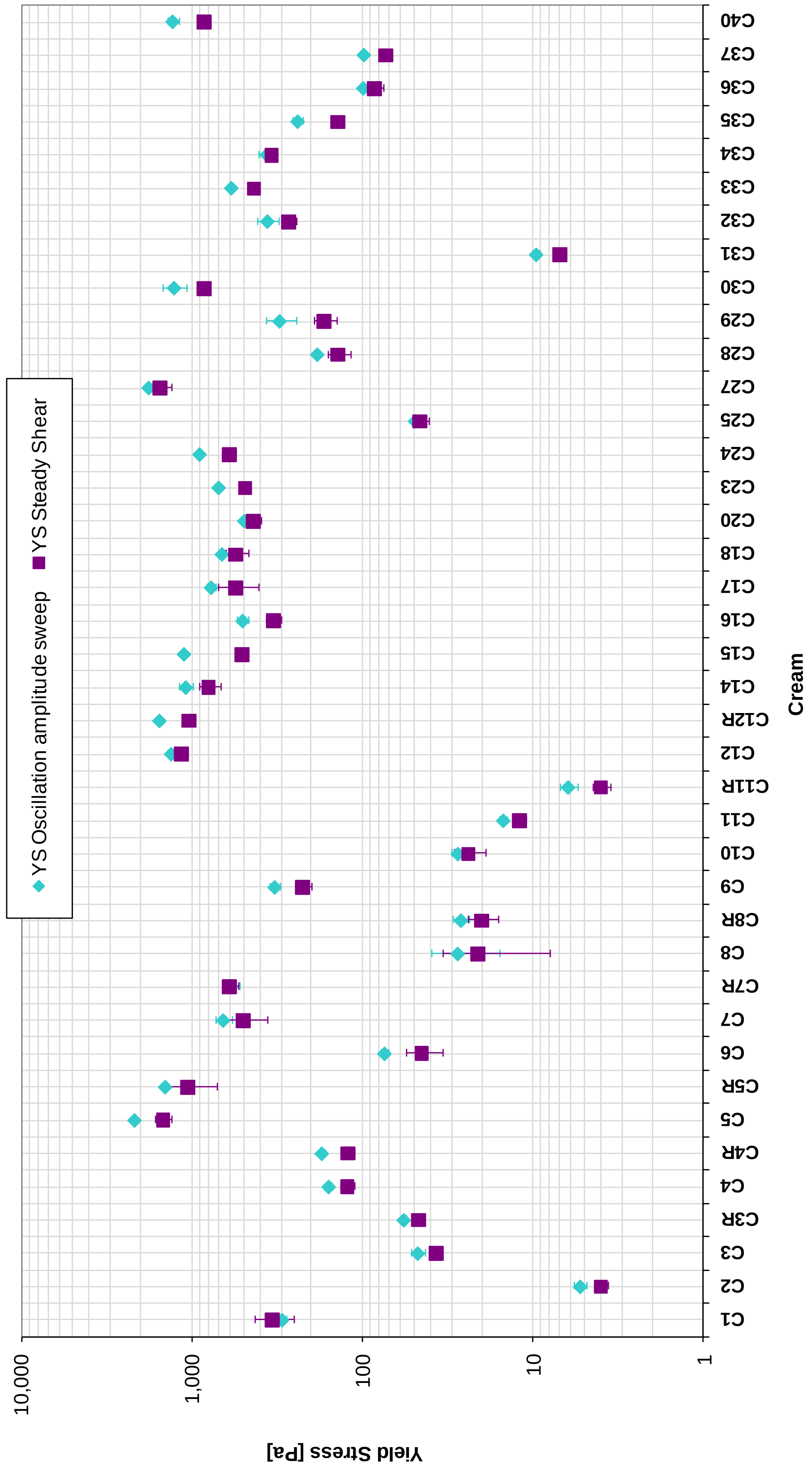


Figure 3.35: *Average results of stress sweeps for three model skin creams: creams 4, 11 and 12R, exhibiting thin to thick behaviour respectively.*

From the flow curves, yield stress values and Cross model parameters were calculated, see Equation (2.2), Chapter 2.4.1.2.1. Results are given in Appendices X and XI, Tables A10.1 - 10.3 and A11.8 – A11.10. Yield stress values were very similar to those derived from the oscillation amplitude sweep see Figure 3.36. This indicates that the different types of measurement and methods for calculating yield stress were complementary.

Figure 3.36: *Comparison between average yield stress results from oscillation amplitude sweep and steady shear (lower end of flow curve) measurements. Error bars determined from SD data are also given.*



The η_∞ values resulting from Cross model calculations showed large errors for several of the creams. This was expected because the flow curves for the majority of the creams 'dropped off' before reaching potential infinite shear viscosities (see example in Chapter 2.4.1.2.1, Figure 2.14). Therefore η_∞ values were not used for comparison between cream samples.

The a-values (Cross time constants) are plotted in Figure 3.37. The reciprocal of the a value (1/a) provides an indication of the shear rate at which shear thinning happens significantly (Cunningham, 2009). Higher a-values therefore relate to samples for which break down occurs at lower rates of shear (Cross, 1965). Samples with limited structure, in particular thinner samples (e.g. C2 and C11), had higher a-values, while those with more structure (e.g. C5 and C25) had lower a-values. Similar information was gained from comparing the yield stress, yield strain and tanδ values obtained through the oscillation amplitude sweep measurements (see Chapter 3.3.1) hence this is not discussed here.

The p-values (also known as Cross rate constants) for the 40 model skin creams are plotted in Figure 3.38. These values indicate the degree by which viscosity depends on shear rate in the shear thinning region (i.e. the slope of the line). A Newtonian fluid would have a p-value of zero (Cunningham, 2009). Results show that p-values range between 0.7 - 1 with thinner samples typically having lower p-values than thicker samples. The extent to which the sample is structured depends on the material response, thinner samples (e.g. C2, C11) have less structure, hence, the viscosity is less dependent on shear rate applied (lower p-values). On the other hand, thicker samples (e.g. C5, C12R) have more structure and therefore shear thinning can occur to a greater extent in these samples (higher p-values). The strength of the material structure, however, is also important in understanding how a material responds under different stresses and strains this information was gained through analysis of yield stress, yield strain and tanδ values as discussed in Chapter 3.3.1.

A comparison between the η^* at 1 % strain and the η_0 is given in Figure 3.39. Results revealed the same trend for both these measurements which could be expected as 1 % strain was within the LVD for most of the samples thus it is a measure of a samples viscosity prior to irreversible deformation. Likewise the zero shear viscosity is measured before irreversible deformation. The Cox-Merz rule is an empirical function that relates the complex viscosity measured in oscillatory shear, $\eta^*(\omega)$, with the apparent viscosity measured in shear flow, $\eta_a(\dot{\gamma})$, (Cox and Merz, 1958; Rehage and Hoffmann, 1988; Kim and Yoo, 2006), the rule applies only when angular frequencies and shear rates are equal:

$$\eta^*(\omega) = \eta_\mathrm{a}(\dot{\gamma})\big|_{\omega=\dot{\gamma}} \qquad (3.4).$$

The Cox-Merz rule does not hold true for suspensions or complex materials unless a shift factor (α) is applied (Gleissle and Hochstein, 2003; Kim and Yoo, 2006):

$$\eta^*(\alpha\omega) = \eta_\mathrm{a}(\dot{\gamma})\big|_{\omega=\dot{\gamma}} \qquad (3.5).$$

Therefore, plotting the complex viscosity results at 1 % strain with viscosity results at 1 s^{-1} (as calculated using the Cross model), see Figure 3.40, revealed that results for the viscosity at 1 s^{-1} are lower than the complex viscosity. In this PhD research it was not possible to calculate a shift factor since the different samples were undergoing rapid shear thinning to different extents at 1 s^{-1}. This also explains why results measured in shear flow were lower than the complex viscosity values at 1 % strain. Therefore in this case the Cox-Merz rule does not apply.

In their research investigating sensory spreadability and the rheological properties operating during application of topical preparations (see Chapter 1.8), Barry and Grace (1972) found that consumers preferred samples with viscosities in the range 0.39 – 1.18 Pa.s where the rate of shear was 400 – 700 s^{-1}. Using the Cross model (see Equation (2.2), Chapter 2.4.1.2.1), viscosities for skin creams used in this research can be calculated at any of the shear rates measured (0.0001 – 10,000 s^{-1}). Therefore the viscosities of the creams at 400 and 700 s^{-1} were calculated. Of the creams used in this study, those with viscosities between 0.39 and 1.18 Pa.s at 400 – 700 s^{-1} were creams 1, 3, 8, 11R and 28. Looking at the consumer study results (see Chapter 3.2), it was interesting to find that vector model consumers in cluster 3 (20 consumers) also preferred these samples (Chapter 3.2.5.1) although other consumer groups showed different liking behaviour. In the study carried out by Barry and Grace, only 10 participants were asked about their preference for the samples used in the study, whereas this research involved 148 consumers hence the wider range of liking behaviours obtained.

Figure 3.37: *Average Cross model a-values for model skin creams as obtained from triplicate steady shear measurements. Error bars determined from SD data are also given.*

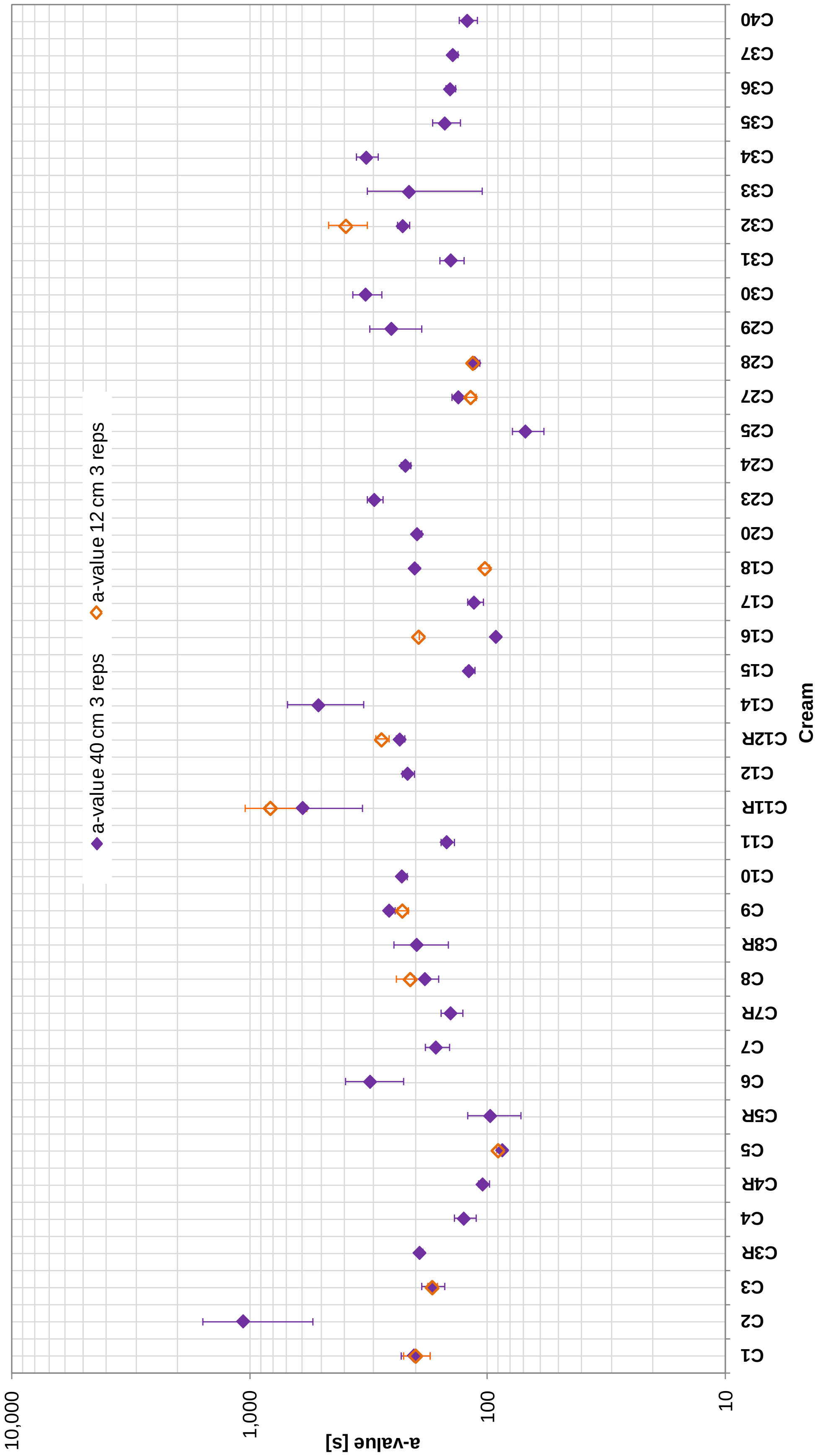


Figure 3.38: *Average Cross model p-values for model skin creams as obtained from triplicate steady shear measurements Error bars from SD data are also given.*

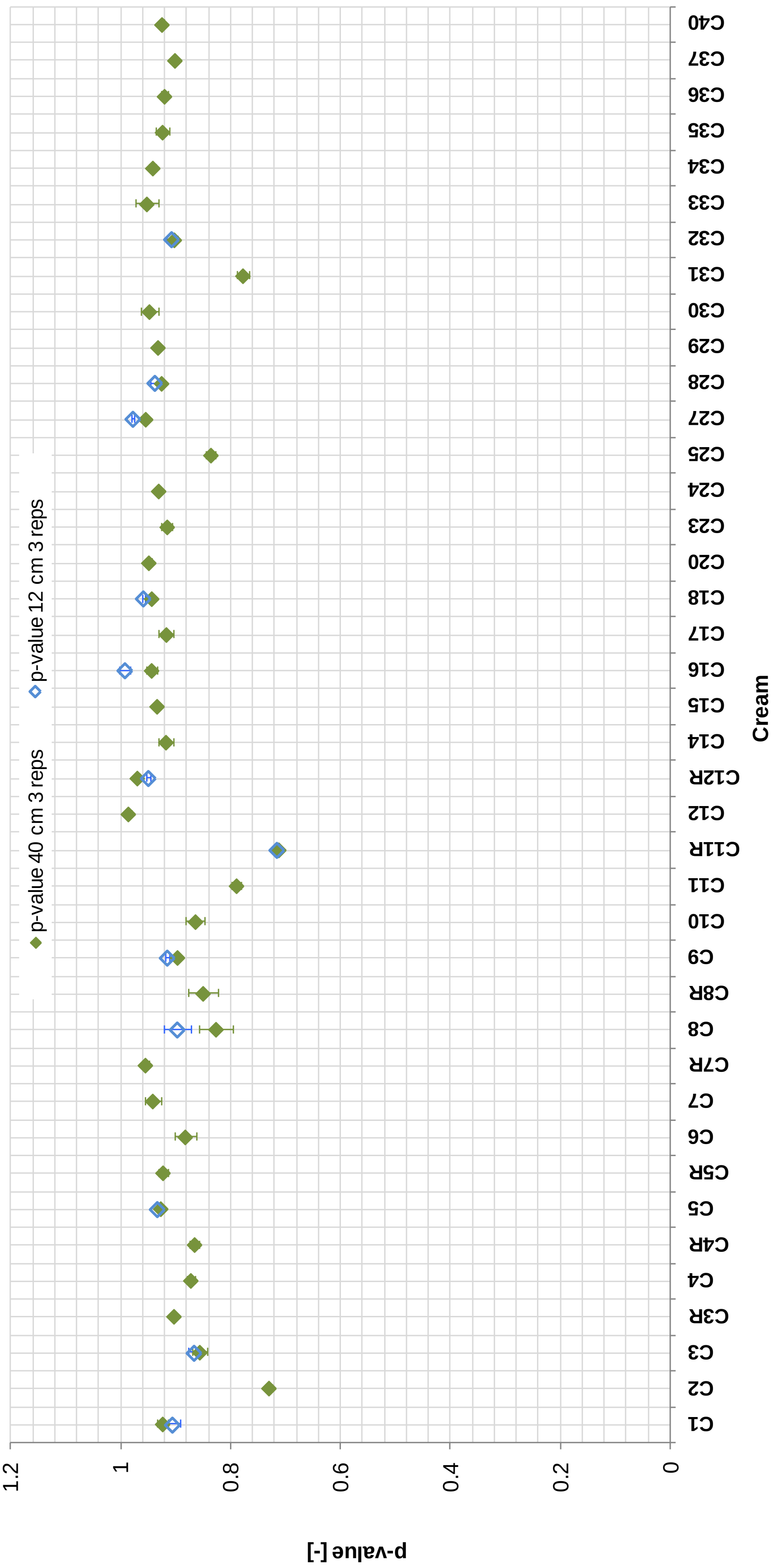


Figure 3.39: *Comparing average zero shear viscosity and complex viscosity for triplicate model skin cream data. Error bars determined from SD data are also given.*

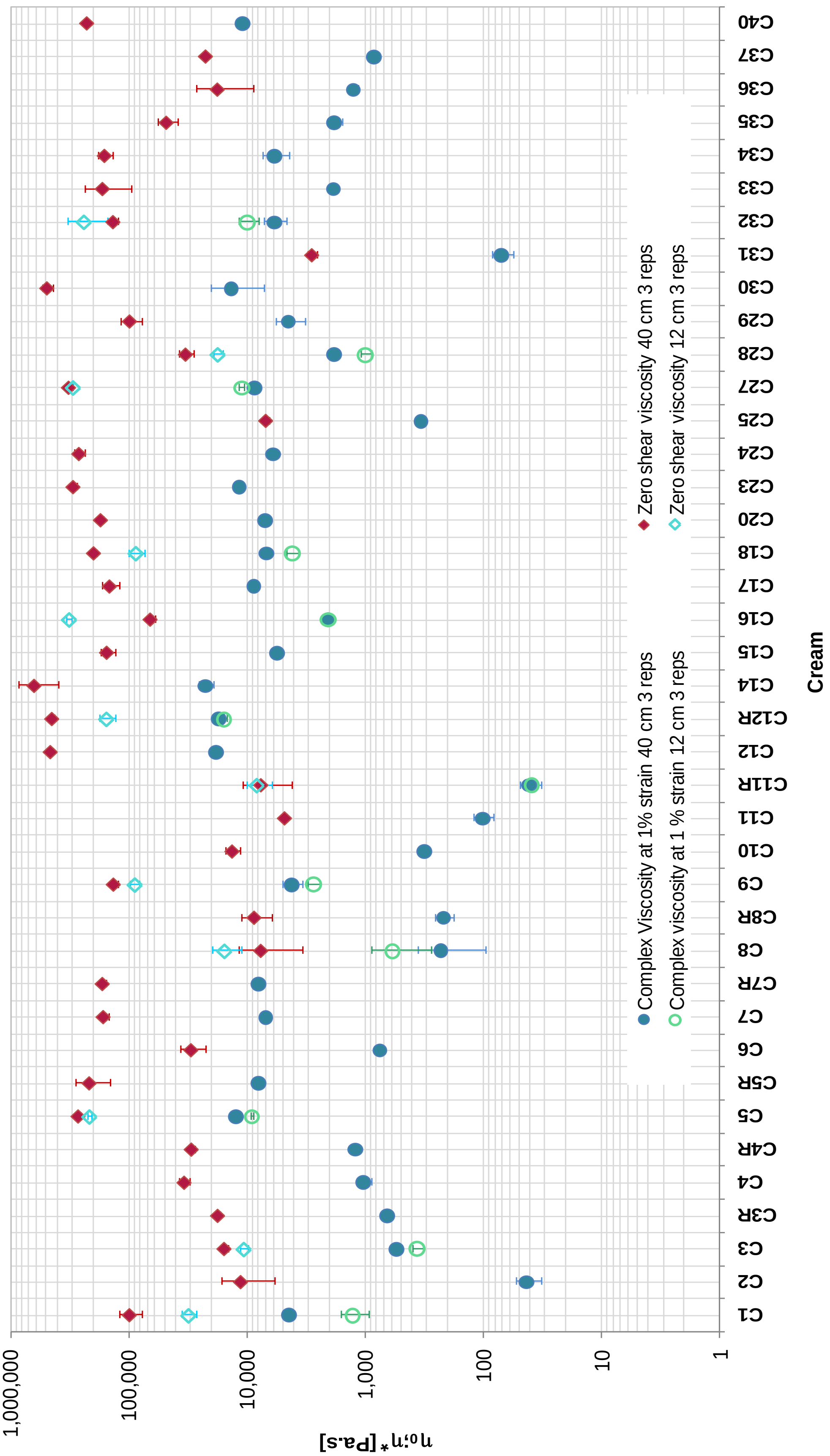
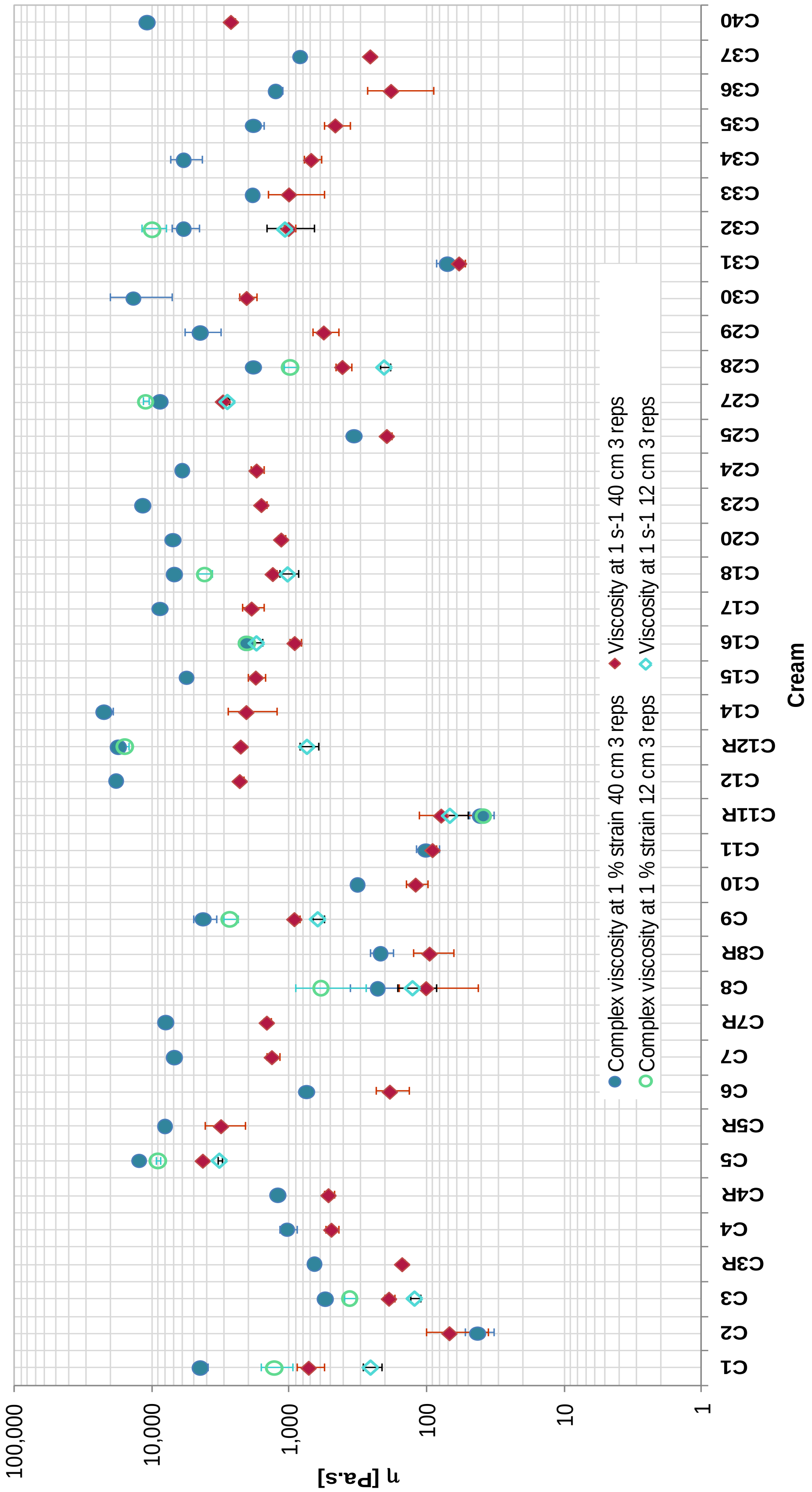


Figure 3.40: *Comparing average viscosity at 1 s^{-1} and complex viscosity at 1 % strain for triplicate model skin cream data. Error bars determined from SD data are also given.*



3.3.4 High shear measurements

In the thin film rheology tests, it was found that in most cases the upper shear rate limit was not reached due to the thinning properties of the creams leading to spinning through of the rotating geometry. Thus any further data-collection was meaningless as mentioned in Chapter 2.4.1.2.2. Therefore, repeat measurements of this test were not taken for the majority of the skin creams and down curve data was not analysed. The viscosity at 10,000 s^{-1} was recorded for each cream, as was the highest useful shear rate value in view of future tests. An example of results for three model cream samples is given in Figure 3.41 (only the 'up-curve' data is shown).

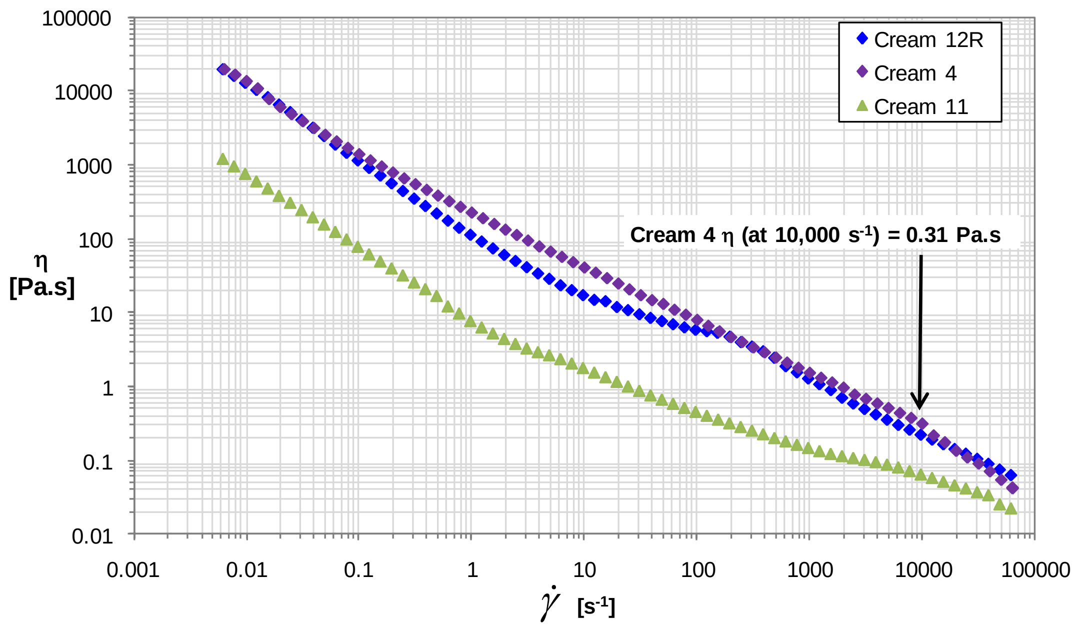


Figure 3.41: *Average thin film rheology results for three model skin creams - creams 4, 11 and 12R from thin to thick respectively.*

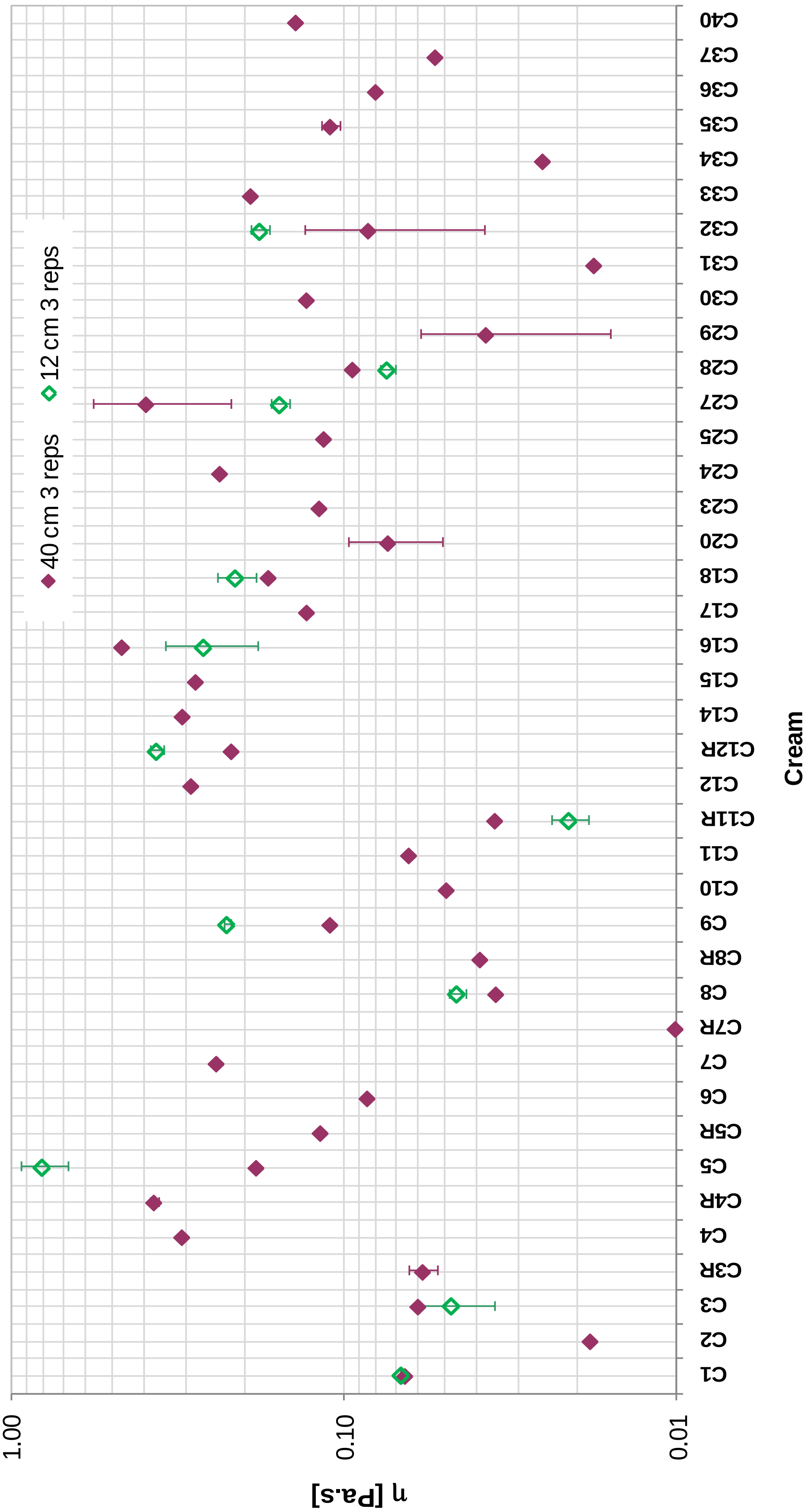
For the majority of samples, reasonable data was collected up to ~60,000 s^{-1}; exceptions were creams 14, 18 and 32. Viscosity results recorded at 10,000 s^{-1} are presented in Figure 3.42, which shows that the viscosities range between 0.01 and 1 Pa.s. Since replicate data was not recorded for most samples when measuring the 40 creams, firm conclusions cannot be drawn from these results.

Triplicate data was however obtained for measurements on the 12 consumer study creams.

If a sample has a high viscosity at 10,000 s^{-1} it is likely to also have a high viscosity at low shear. All the samples with viscosities > 0.2 Pa.s at 10,000 s^{-1} had complex viscosities > 1000 Pa.s at 1 % strain. It is however interesting that cream 4 and cream 12R have similar viscosities at high shear (C4 = 0.308 Pa.s and C12R = 0.219 Pa.s) whereas at low shear they were quite different (C4 =1037 Pa.s and C12R = 17450 Pa.s). This again provides information about the structure of the samples. Cream 4 has a stronger internal structure as illustrated by tanδ < 1 at 100 % strain (see Figure 3.32, Chapter 3.3.1), whereas cream 12R has a weaker structure with a tanδ > 1 at 100 % strain indicating that this sample breaks down more rapidly under shear than cream 4.

Creams with low viscosities at high shear should be easier to apply to the skin (see Chapter 1.5.2.1). Comparing viscosities measured at 10,000 s^{-1} for the consumer study creams with sensory QDA scores for spreadability, revealed that samples with lower viscosities at high shear (< 0.1 Pa.s) were indeed easy to spread (QDA spreadability scores < 3.5).

Figure 3.42: *Viscosity results for the skin creams obtained in thin film rheology tests as measured at 10,000 s^{-1}.*



3.3.5 Summary

The rheological characterisation of the skin cream samples emphasised the range of properties encompassed by the 40 model skin creams used in this PhD. Results provided useful information about the behaviour of the skin creams under different conditions (shear and strain controlled) which formed the basis for understanding the relationships between sensory attributes and rheological parameters as discussed in Chapter 4.

Overall the oscillation amplitude sweep and the low shear measurements provided the most useful information regarding cream behaviour under different stresses. The majority of information gained from analysing Cross model parameters from the steady shear measurements could also be gained through analysis of oscillation amplitude sweep data. However, the Cross model equation was useful for calculating viscosities at specific shear rates and comparing with data from other studies. Frequency sweeps and high shear data did not add any further information to that gained from the low shear and oscillation amplitude sweeps. Therefore the oscillation amplitude sweep would be the measurement of choice if further research into skin creams were to be carried out and then if time permitted, the low shear measurements would also be recommended.

3.4 TEXTURE ANALYSIS

Typical curves obtained in the back extrusion measurements on the model skin cream samples (as described in Chapter 2.4.2) are given in Figure 3.43. The three data sets shown illustrate the range of curves obtained for thin to thick cream samples, error bars are included giving the standard deviations calculated from six replicate measurements.

In general, good replicate data was obtained, however, for the thicker samples in some cases error was observed in the negative curve as can be seen in Figure 3.43 for cream 5 (black shading). The cause of error is likely to be due to the forces acting on the probe as it was lifted out of the sample. These forces were larger for thicker samples and the probe could not be removed easily from the sample. This created a lot of pressure on the container, which was hand held in position. It is possible that in some cases the container moved slightly which may be the cause of the larger error as observed in the case of cream 5.

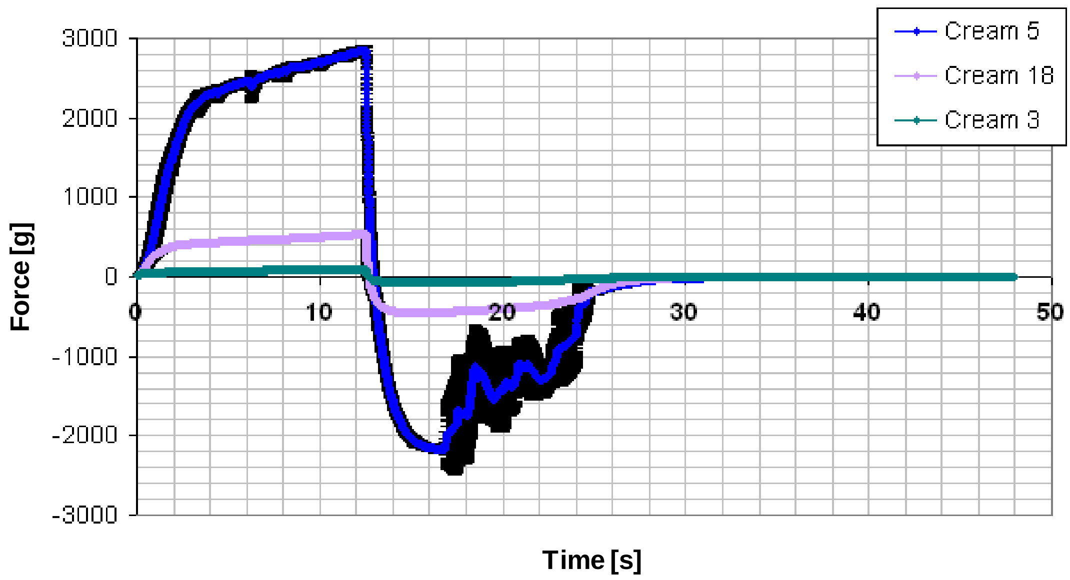


Figure 3.43: *Typical plots from back extrusion illustrating the behaviour of three samples, creams 3, 18 and 5 from thin to thick respectively.*

Average results for firmness (maximum positive force reading), consistency (area under positive curve), cohesiveness (maximum negative force reading) and index of viscosity (area under negative curve) were evaluated for each cream (see Chapter 2.4.2). Results are given in Figures 3.44 - 3.47. Creams 5 and 27 were the firmest samples with the highest consistency values reflecting the fact that these two creams were the thickest samples. Creams 11R and 3 showed the lowest values for both these parameters suggesting they were the thinnest samples. The same trend was observed looking at results for cohesiveness and index of viscosity whereby creams 5 and 27 had the most negative values indicating they were the most cohesive and most viscous samples. Equally creams 11R and 3 had the least negative values indicating they were the least cohesive and least viscous samples. The range of values found was considerably large which could be expected given the choice of samples in this set (see Chapter 3.2.1).

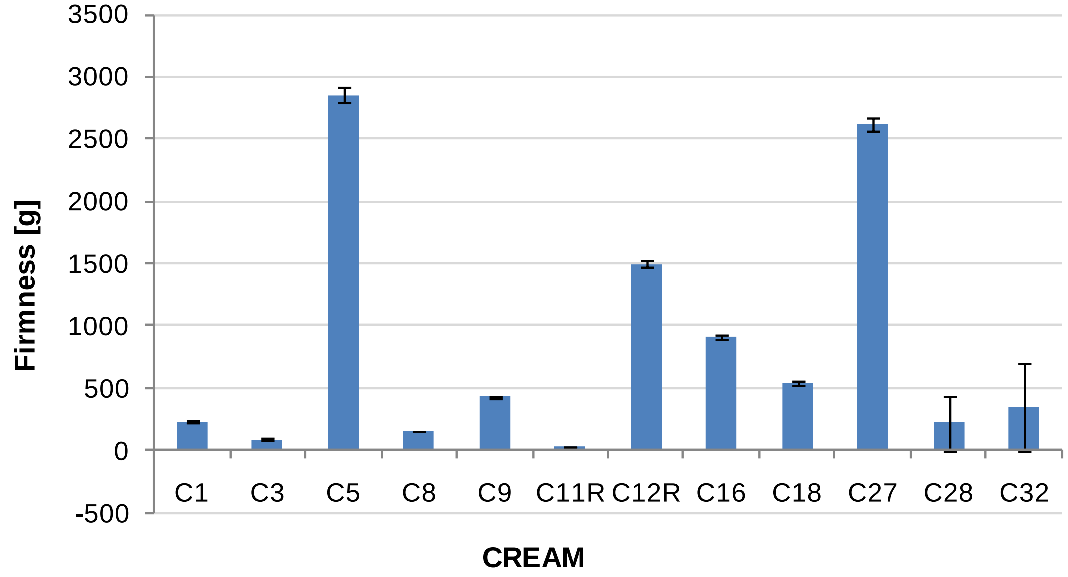


Figure 3.44: *Average firmness results as obtained by back extrusion for the 12 consumer study creams. Error bars determined from SD data are also given.*

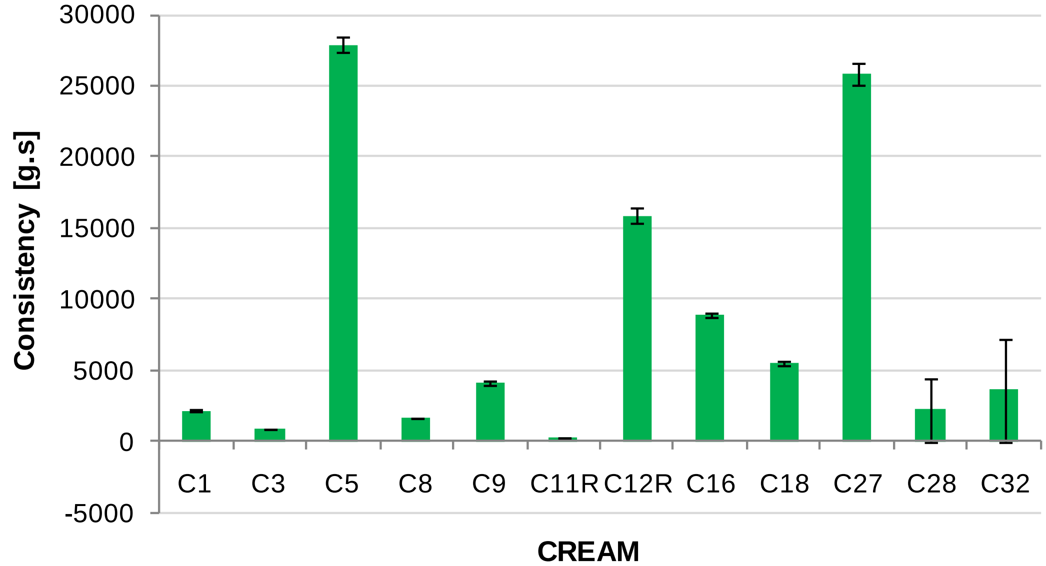


Figure 3.45: *Average consistency results as obtained by back extrusion for the 12 consumer study creams. Error bars determined from SD data are also given.*

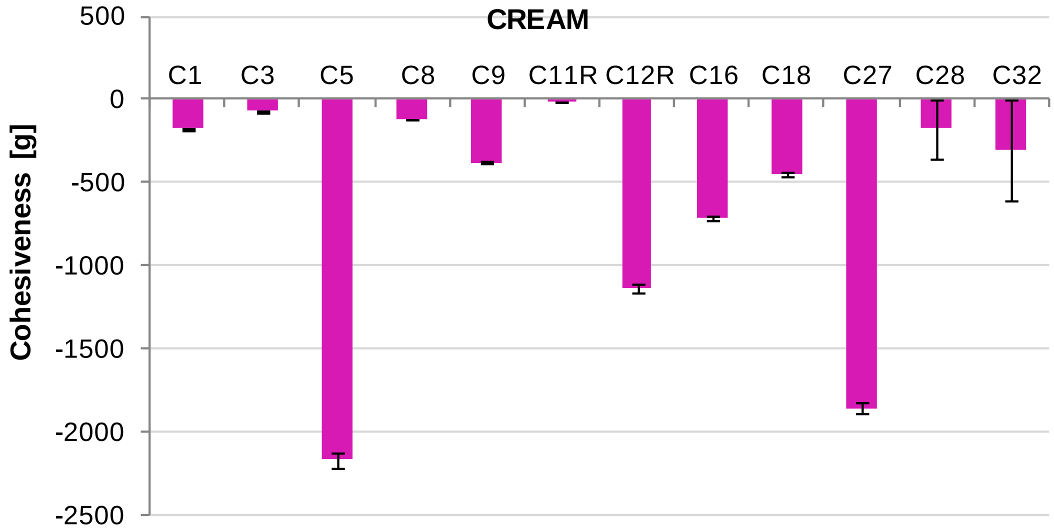


Figure 3.46: *Average cohesiveness results as obtained by back extrusion for the 12 consumer study creams. Error bars determined from SD data are also given.*

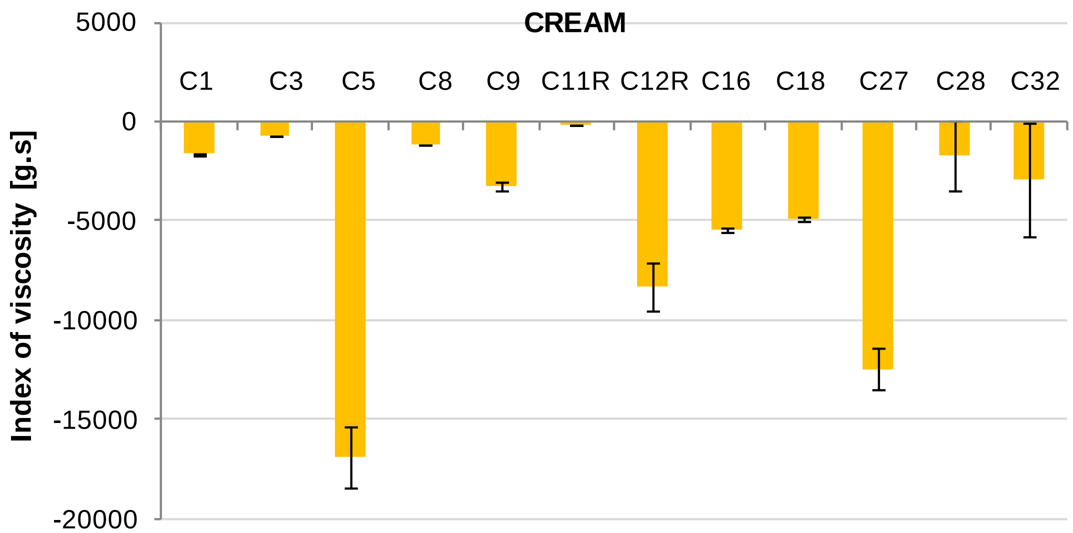


Figure 3.47: *Average index of viscosity results as obtained by back extrusion for the 12 consumer study creams. Error bars determined from SD data are also given.*

Results obtained from back extrusion provide an indication of how the cream will behave in different packaging types and during processing (see Chapter 1.6.2). In this case creams 11R and 3 have the lowest values for consistency, firmness, index of viscosity and cohesiveness. This suggests that they could be easily pumped around a factory and may therefore be more suited to a pump style packaging as a container may result in spillages. On the other hand creams 5 and 27 have the greatest consistency therefore they may be unsuitable for pumping through a factory and they would need to be delivered to the consumer in a container as they may be too thick to extrude through a tube.

The cohesiveness and index of viscosity data are also thought to influence the spreading properties of the sample. Comparing the rank order of the cohesiveness data with the spreadability has revealed a similar trend. The extreme samples, creams 11R, 3 (easiest to spread), 12R, 5 and 27 (the most difficult to spread) were also the least and most cohesive samples respectively. This is understandable as the more cohesive a sample, the stronger the interaction between molecules in the structure is, thus the harder the sample will be to spread on the skin.

Results obtained through back extrusion showed the same trend as yield stress data obtained through rheological measurements. This makes sense as the back extrusion test measures the forces involved in deforming the sample while the yield stress is a measure of the force required to induce flow in a product (see Chapter 1.5.2.2). Tamburic et al. (1996) used a texture analysis penetration test to measure the properties of different skin cream formulations. The penetration test measured the work of cohesion and adhesion experienced by the sample. In their study they found that the rank order of cohesiveness and adhesiveness followed a similar trend as was the case in this study (firmness, cohesiveness, consistency and index of viscosity all followed same trend). However, Tamburic et al. (1996) found that cohesiveness and adhesiveness results were correlated to viscosity values obtained at 50 Pa (the highest stress measured), following a slightly different trend to the yield value which was also measured. The difference between Tamburic's results and this study is likely to be related to the different type of test carried out. Penetration tests involve the use of different shaped probes to that used in back extrusion, see Figure 3.48.

Figure 3.48: *Shapes of probes used in penetration and back extrusion tests*

Tamburic et al. (1996) used a 13 mm diameter probe whereas in this study the probe was 38 mm in diameter. The smaller diameter of the probe used in the penetration test would have created a greater force acting on the sample hence the relation of the penetration results to viscosity values obtained at high shear. On the other hand the wider probe diameter used in the back extrusion test will have created less force on the sample allowing for forces involved in deforming the sample to be measured hence the correlation of back extrusion results with yield stress values.

As mentioned in Chapter 1.6.2, the firmness measured through back extrusion may be related to the sensory firmness observed when a consumer dips their finger into a sample of cream. In this study, the trained panel measured the firmness by dipping their index finger in the sample and observing the resistance of the cream to movement (see Table 2.3, Chapter 2.3.2.3). Looking at the trained panel results for firmness (see Table 3.5, Chapter 3.2.2), reveals that exactly the same rank order of firmness was obtained by the trained panel as that obtained by the texture analyser. This suggests that the firmness obtained through back extrusion measurements may be a useful parameter in the generation of predictive models.

3.4.3 Summary

Texture analysis was used to characterise the consumer study creams for the parameters firmness, consistency, cohesiveness and index of viscosity. Results followed the same rank order of discrimination between the creams for the different parameters. Creams 5 and 27 were the thickest, firmest samples with the greatest cohesiveness and resistance to flow while creams 3 and 11R were the thinnest, least firm creams with the lowest cohesiveness and resistance to flow. Yield stress data from rheological measurements and sensory firmness data showed the same trend as the results from back extrusion while a similar trend between sensory spreadability and back extrusion data was also found.

3.5 FORCE PLATE ANALYSIS

The relative humidity (RH) and temperature of the room in which force plate analysis was conducted remained relatively constant throughout measurements: 51 % RH (± 2 %) and 22.5 °C (± 1 °C) respectively. The effects of load and speed on friction coefficient or friction were compared for various samples. It is not practical to discuss results from all episodes for all cream samples in this chapter, therefore results discussed here were taken from episode 6 for four samples, creams 27, 28, 3 and 11R from thick to thin respectively.

The relationship between friction and load is illustrated in Figure 3.49. It is clear that increasing the load results in an increase in friction for all samples. Cream 11R had the lowest friction readings throughout the measurement, which suggests it was a slippery, greasy cream that formed a more lubricating layer between the fingertip and Bioskin than the other samples. The lower the friction the more slippery the cream is expected to be (see Chapter 2.4.3.1). Trained panel results reveal slipperiness scores of 2.0, 8.5, 9.1 and 9.2 for creams 27, 3, 11R and 28 respectively (see Table 3.7, Chapter 3.2.2), thus agreeing with the theory that slippery samples result in lower friction between surfaces. Creams 3 and 27 showed the highest friction readings with increasing load, suggesting there was limited lubrication between the surfaces either due to the cream having already absorbed leaving dry surfaces behind or the creams being drying in nature. Trained panel results confirm that creams 27 and 3 were samples with relatively high drying properties with drying scores of 6.5 and 7.4 respectively, compared to 0.3 and 2.5 for creams 28 and 11R respectively (see Table 3.6, Chapter 3.2.2).

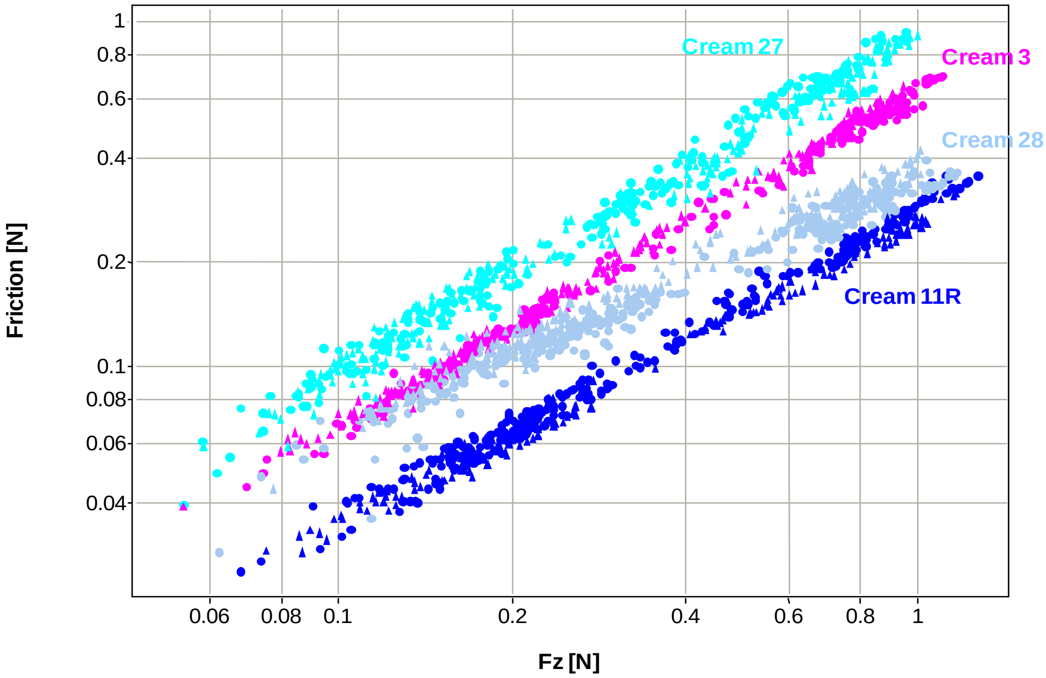


Figure 3.49: *Relationship between friction and load (Fz) for four consumer study creams as collected during episode 6 where the speed range is 50 - 100 mm.s^{-1}. Replicate data points are indicated by circles and triangles respectively.*

The extent by which friction increased depended on the sample. For example, in the case of cream 28, friction increased more slowly with load than it did for the other samples (Figure 3.49). This behaviour is typical of a thick cream where at high loads the friction is relatively low as the cream provides good lubrication because it resists being squeezed out of the gap. Yet at lower loads the friction is relatively high because of a high yield stress creating fluid drag (see Chapter 1.7.1). Note that of the four samples illustrated in Figure 3.49, cream 27 was the thickest when in the fresh form (see Appendix XI, Table A11.2 and A11.8). However, this chapter discusses results obtained during episode 6 where the sample has undergone a high level of stroking deformation and is therefore present in a thin layer. The results in Figure 3.29 therefore show that cream 28 is more lubricating following high levels of stroking deformation than cream 27, thus suggesting that cream 28 is greasier. Trained panel results confirm that cream 28 is a lot greasier than cream 27 with final greasiness scores of 9.1 and 2.1 respectively.

Loden et al. (1992) found that the frictional resistance of a cream during application depended on the viscosity (as measured with a Haake Viscometer at 20 °C, load 0.1 N); the higher the viscosity, the higher the frictional resistance. The rank order for the creams in Figure 3.48 for η^* (at 1 % and 100 % strain) and η_0 from highest to lowest follows: 27, 28, 3, 11R (see Tables A11.2, A11.3 and A11.8, Appendix XI). Therefore similar findings were observed in this research when comparing the viscosity of the samples with the friction at 0.1 N (the load used by Loden et al.). However, at high loads, the order of creams 28 and 3 differs. This suggests that for cream 28, the friction between surfaces depends on load applied.

Loden et al. (1992) also found that a higher greasiness was related to a higher oil content and lower friction. Similar findings were found by Nacht et al. (1981). Likewise in this research, samples with higher oil content, (cream 11R = 20 % oil and cream 28 = 40 % oil) had lower friction values than the samples containing no oil (creams 3 and 27). In agreement with Loden et al. (1992) and Nacht et al. (1981) this was also related to the final greasiness although in this case the relationship was not linear. Final greasiness scores follow: C28 = 9.1, C11R = 5, C27 = 1.2 and C3 = 0.2. Results plotted in Figure 3.49 show that friction was proportional to load for most samples thus samples were following Amontons' law (see Chapter 1.7.1). It is therefore more useful to plot the friction coefficient (friction/Fz) against Fz as the friction coefficient is generally independent of load (Asserin et al., 2000). This form of plot is also more useful for statistical analysis i.e. calculating the log(Fz) factor (see Chapter 2.4.3.2).

The relationship between friction coefficient and load for the same four samples is given in Figure 3.50. Results show that the friction coefficient for creams 3, 11R and 27 was independent of load. The negative slope seen for cream 28 suggests that the friction coefficient was weakly dependent on load as could also be seen by the less steep slope in the friction versus load plot (Figure 3.49). This suggests that the film formed by cream 28 between the fingertip and bioskin is thick enough during episode 6 to determine the frictional properties between the surfaces rather than Amontons' laws holding true (see Chapter 1.7.1).

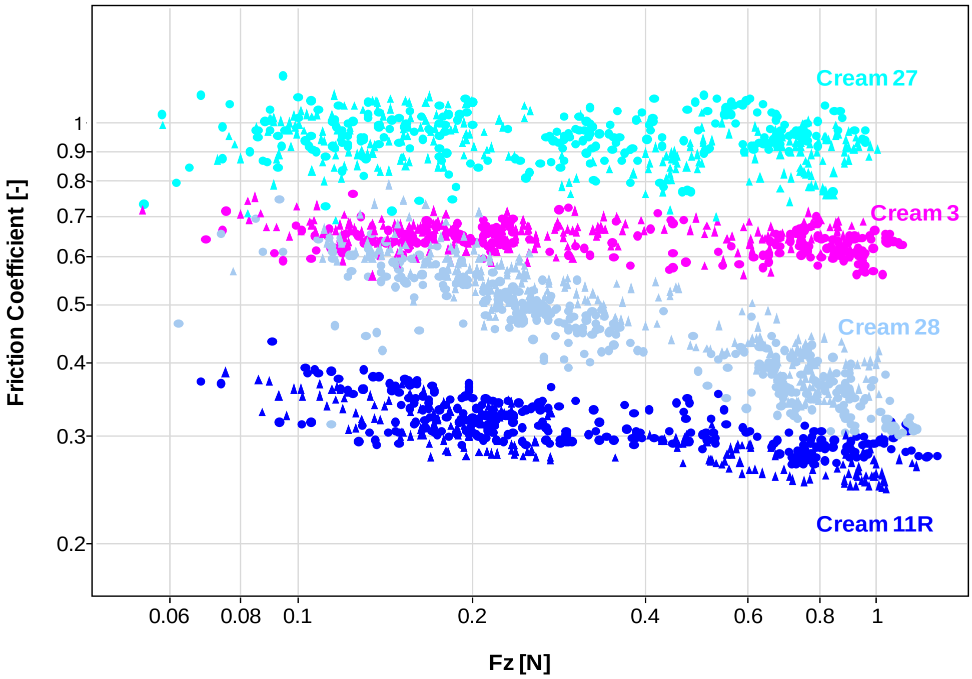


Figure 3.50: *Relationship between friction coefficient (friction/Fz) and load (Fz) for four consumer study. Data relates to episode 6, speed range 50 - 100 mm.s^{-1}. Replicate data points are represented by triangles and circles respectively.*

In their study into the relationship between touch perception and surface physical properties, Chen et al. (2009) found that the friction coefficient (μ) was related to the following pairs of adjectives (these were scale anchors as rated by panellists in their study): slippery - sticky and wet - dry where a high μ was related to a stickier surface and a low μ was related to a drier surface. Comparing the μ results from this research with sensory QDA scores (see Chapter 3.2.2, Table 3.7) revealed that a high stickiness score was related to a high μ although in this case, a cream with a high drying score was related to a high μ thus opposite relationship to that found by Chen et al. (2009). However, it must be noted that the work carried out by Chen et al. was in relation to packaging types (thus dry surfaces) while this work is looking at lubricated films. The presence of a lubricated film is reported to increase the μ between surfaces (Prall, 1973; Nacht et al., 1981), hence the difference between the two studies. Also, during episode 6, cream samples that are drying will be present in thinner layers thus allowing a high contact area between surfaces resulting in the high friction coefficient values (see Chapter 1.7.1).

The relationship between friction and speed revealed that an increase in speed led to an increase in friction except for cream 11R (see Figure 3.51). This observation is related to the viscosity of the samples. High speeds lead to an increase in friction. This is particularly prevalent in more viscous samples due to fluid drag (See Chapter 1.7.1). If the cream is thin, less force is required to deform the sample (lower yield stress) therefore at high stroking speeds, the friction will be relatively low compared to a thicker sample (this only applies if there is a complete fluid film between surfaces i.e. no surface-surface contact). The complex viscosities of these creams at 100 % strain follow: 7 Pa (C11R), 50 Pa (C3), 157 Pa (C28) and 2430 Pa (C27). In this case the most and least viscous samples have the highest and lowest friction coefficients respectively. However, cream 3 has a low viscosity yet a high friction. This suggests after high levels of stroking deformation, cream 3 can no longer form a complete fluid film between the surfaces due to drying out. Therefore in this case the contact between the two surfaces where layers of cream are not present results in the higher friction readings. Comparing the drying properties of the samples reveals that cream 3 is the most drying sample with a drying score of 7.4 compared to 0.3, 2.5 and 6.5 for creams 28, 11R and 27 respectively.

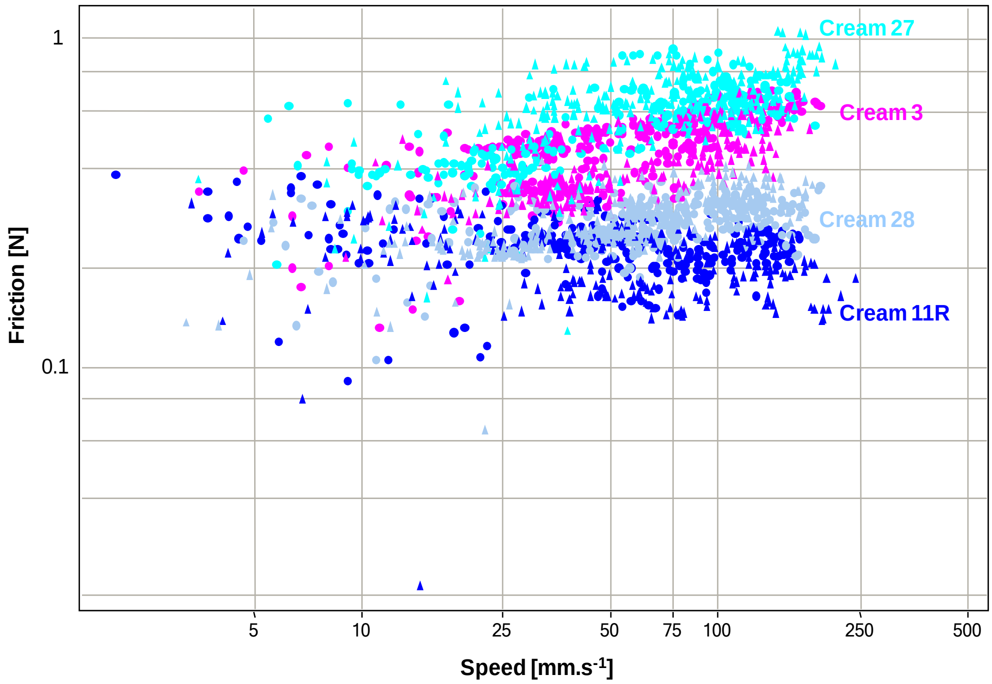


Figure 3.51: *Relationship between speed and friction for four consumer study creams Data relates to episode 6, load range 0.5 - 1 N. Replicate data points are represented by triangles and circles respectively.*

Plotting the friction coefficient (friction/load) against speed (Figure 3.52) infers a different relationship for cream 11R, which has a slight decrease in friction coefficient with increase in speed. This suggests that cream 11R becomes more lubricating at higher speeds, while the other samples show a decrease in their lubricating capacity. This could be due to syneresis, which can occur if the cream formulation is not stable i.e. it separates becoming more lubricating at higher speeds. Interestingly, during sensory panel training for the attribute final greasiness, it was discovered that certain samples absorbed and then with further spreading on the skin, the greasy residue returned. Cream 11R was such a cream and this characteristic behaviour is represented in the force plate results presented in Figure 3.52, hence the two test methods are in agreement.

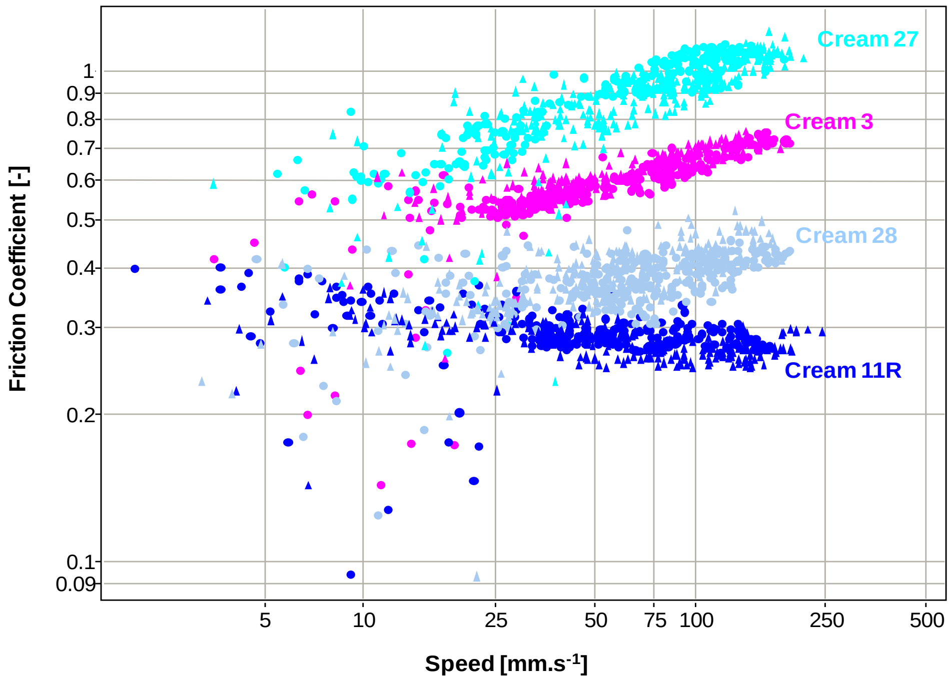


Figure 3.52: *Relationship between speed and friction coefficient for four consumer study creams Data relates to episode 6, load range 0.5 - 1 N. Replicate data points are represented by triangles and circles respectively.*

The Stribeck curves for the same creams are given in Figure 3.53. The slightly positive slopes observed for creams 3, 27 and 28 suggest the lubrication between surfaces is in the hydrodynamic regime (see Chapter 1.7.1). Cream 11R appears to show a shallow dip in friction coefficient around a speed/Fz of 100. This suggests it is in the mixed or EHL regime. Previously discussed relationships showed a decrease in friction coefficient with speed, which agrees with this observation (see also Figure 1.4, Chapter 1.7.1).

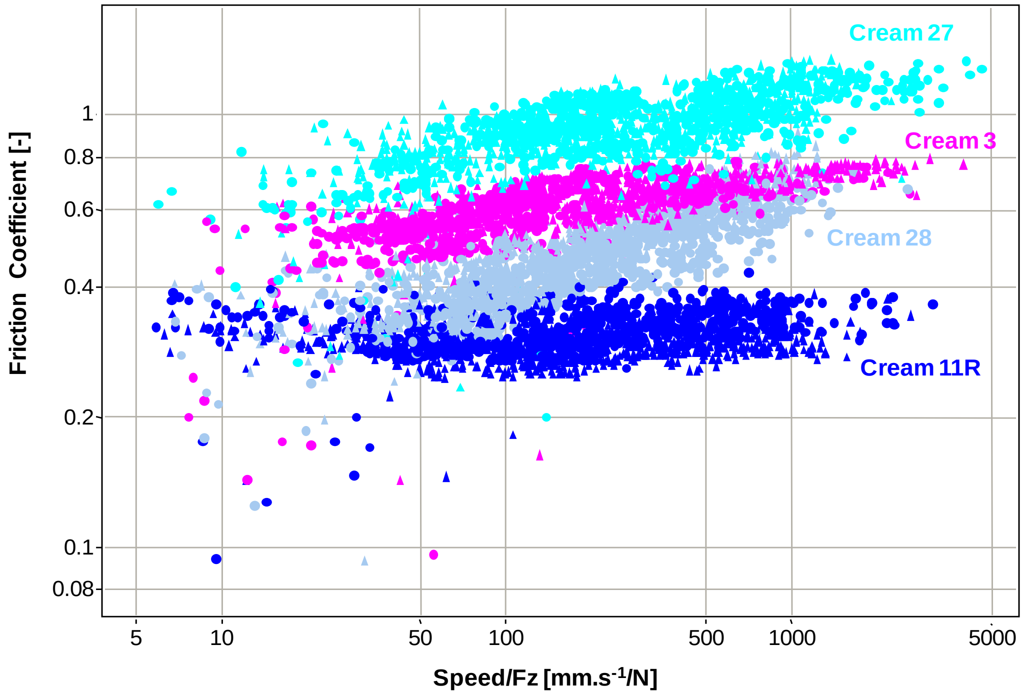


Figure 3.53: *Relationship between speed/Fz and friction coefficient for four consumer study creams. Data for episode 6, all load speed ranges are given.*

3.5.1 Summary

Force plate results were plotted in graphs of friction or coefficient against load and speed. Visualisation of results in this manner allowed a greater understanding of the cream properties involved in this study to be gained. Of the four creams considered in this section, it was found that cream 27 showed the highest friction readings throughout, which suggests it was the least slippery (or least lubricating) of the samples. Cream 3 showed similar properties to cream 27 although to a lesser extent (slightly more slippery/lubricating than cream 27). Cream 11R appeared to be the slipperiest (most lubricating) sample closely followed by cream 28.

The results compared in this section were all from episode 6 of stroking measurements. This was the last episode from the stroking tests so the samples would have been in their driest state. In Chapter 4 results from earlier episodes and their relationship with the sensory properties of the skin creams will be considered.

3.6 CONCLUSION

QDA and physical measurements were carried out on the 40 model skin creams and subsequently on the fresh batch of 12 consumer study creams to gain quantitative data regarding the properties of the model skin creams. In general, results showed good correlation between data from 40 model skin creams and the 12 consumer study creams ($r = 0.9$). Across most measurements on consumer study creams (TA, rheology, sensory QDA), creams 5 and 27 were identified as the firmest, thickest samples and creams 3 and 11R as the thinnest (out of the 40 model skin creams, creams 5 and 27 were the thickest but creams 10, 31 and 2 were thinner than cream 3). Overall, agreement between physical and sensory results was found regarding properties of the model skin creams.

Consumer study results provided an indication of creams liked by different groups of consumers. Liking was related to the sensory properties of creams with the firmness and thickness standing out as attributes of great importance regarding consumer liking. In Chapter 4 predictive models are discussed whereby sensory properties of model skin creams are predicted from physical parameters. Understanding which attributes are important to the consumer provides an indication of models that will be more useful in terms of predicting attributes that affect consumer liking.

4. RELATIONSHIP BETWEEN SENSORY AND PHYSICAL DATA

The ultimate goal of this research was to be able to predict the sensory properties of a cream from its physical characteristics and to determine how much consumers would like the resulting cream. Principal component analysis (PCA) and predictive modelling were used to determine if this was possible.

Samples

In this chapter average results from analysis of the 12 consumer study creams are discussed since they represent the wide range of creams produced by the initial experimental design and all physical measurements were carried out on these samples. Data from the 40 model skin creams were used to check validation of the rheological models where possible.

Methods

Principal component analysis with varimax rotation *(XLSTAT, Version 2007.6)* was used to visualise the relationship between the physical parameters (measured by rheology, texture analysis and force plate analysis) and the sensory attributes (measured by QDA) for the model skin creams. The aim was to find a few parameters that were closely correlated to attributes on the two PCA axes so predictive modelling based on these parameters could be carried out.

Polynomial predictive regression models were created using average panel scores and average physical parameter data (Design Expert software, version 6.0.2, 2000). These models enabled the sensory properties of the creams to be described in terms of the physical parameters. Non-significant terms as determined by ANOVA were removed and final mathematical models were chosen that best represented the data after scrutiny of best fit equations and associated model values. The ability of the final model to explain the data was indicated by high adjusted R^2 and predicted R^2 values.

R^2 values provide a measure of the variation about the mean explained by the model. These values are calculated using the following equation:

$$\mathbf{R^2 = 1 - (SSresidual\ /\ (SSmodel - SSresidual))} \tag{4.1}$$

where SSresidual is the sum of squares (SS) for the residual, the residual is the portion of the corrected total SS not explained by the model; SSmodel is the sum of the squared residuals for terms in the model (Design-Expert, 2000).

The adjusted R^2 values are similar to the R^2 values but they are adjusted depending on the number of terms in the model; if terms involved in the model add little value to the model the adjusted R^2 values will decrease, see equation 4.2:

$$\mathbf{Adjusted\ R^2 = 1 - ((SSresidual\ /\ DFresidual)\ /\ ((SSmodel + SSresidual)\ /\ (DFmodel + DFresidual)))} \tag{4.2}$$

DFmodel is the degrees of freedom for the selected model i.e. the number of model parameters, including the intercept (if present) minus 1. DFresidual is the degrees of freedom associated with the residual. This number is equal to the corrected total DF minus the model DF (Design-Expert, 2000).

Predicted R^2 values measure how well the model predicts a response value as expressed by:

$$\mathbf{Predicted\ R^2 = 1 - (PRESS\ /\ SStotal)} \tag{4.3}$$

where PRESS is the predicted residual sum of squares for the model which provides an indication of how well the model fits each point in the design. The PRESS is calculated by first predicting where each point should be from a model that contains all other points except the one in question, the squared residuals (difference between actual and predicted values) are then summed. SStotal is the total sum of squares of the model. Predicted and adjusted R^2 values should be within 0.2 of each other to show that the models are able to make reasonable predictions (Design-Expert, 2000).

The final model equations allow sensory properties of samples to be predicted when values for certain physical parameters are known. Predicted values were plotted against actual values (mean scores obtained for the attributes through QDA by the trained panel). These plots included x = y lines allowing residual error to be visualised.

4.1 PRINCIPAL COMPONENT ANALYSIS

Initial PCA highlighted any parameters that did not correlate with the first two principal components (the first and second principal components explained 91 % of variation in textural properties, see Chapter 3.1.2). These parameters were removed from further analysis. They included: yield strain, G'' at 0.1 % strain, $\tan\delta$ at 0.1 %, 1 % and 100 % strain (amplitude sweep); $\log G'$-$\log\omega$ slope, $\log G''$-$\log\omega$ slope, $\tan\delta$ at 1 rad.s^{-1} (frequency sweep), η_∞, a-value, p-value (steady shear); log(Coeff) factor episodes 2 – 6, log(Fz) factor episodes 1 – 2 and log(Speed) factor episodes 1 and 3 – 6 (force plate analysis).

Of the parameters that remained, those that were highly correlated were compared and only one of each group/pair of highly correlated parameters was included in the final analysis. For example rheological analysis showed that the G' and G'' at 1 rad.s^{-1} (frequency sweep) were highly correlated to the G' and G'' at 1 % strain (amplitude sweep) so in this case the data at 1 rad.s^{-1} was removed (see Chapter 3.3.2). Likewise the η^* values were highly correlated to G' values at equivalent low strains (see Equations (3.1 – 3.3), Chapter 3.3.1) so η^* values were removed from further analysis as they were accounted for in the G' data. G' and G'' at 0.1 % strain and at 1 % strain were also highly correlated since they were still in the LVD (see Chapter 3.3.1) therefore only data at 1 % and 100 % strain were included in the models. Yield stress (YS) data from oscillation amplitude sweep (OAS) and steady shear measurements were also highly correlated as expected (see Figure 3.36, Chapter 3.3.3). The OAS YS was chosen for inclusion in the models since the majority of other rheological parameters that fitted with PC1 and 2 were from oscillatory measurements and for the practicality of predictive modelling it would be convenient if all parameters could be extracted from one test.

All texture analysis parameters were included in the PCA. Note that the index of viscosity and cohesiveness data were included as negative values in the PCA analysis and predictive modelling. In the case of force plate parameters the stribeck slopes were not included in PCA since they incorporate both the speed and load factors which were already included as separate terms (see Chapter 2.4.3.2). Table 4.1 provides a list of the final parameters included in the PCA analysis.

Table 4.1: *Physical parameters included in PCA.*

Measurement	Final parameters included in PCA
RHEOLOGY	
Amplitude sweep	• G′ & G″ at 1 % and 100 % strain • Yield stress *(annotated as OAS YS in the PCA plot)*
Frequency sweep	• logG′ - logω intercept • logG″ - logω intercept
Thin film rheology (high shear measurements)	• η at 1000 s^{-1} • η at 10,000 s^{-1}
Stress controlled (low shear measurements)	• η_0
TEXTURE ANALYSIS	
Back extrusion	• Firmness • Consistency • Index of viscosity • Cohesiveness
FORCE PLATE ANALYSIS	
Friction measurements	• Coefficient factor at 100 $mm.s^{-1}$ and 0.5 N, episode 1 (log(Coeff) Factor E1) • Speed factor episode 2 (log(Speed) Factor E2) • Load factor episodes 3, 4, 5 & 6 (log(Fz) Factor E3 - 6)

Figure 4.1 illustrates the relationship between the sensory attributes and the physical parameters. Results from this PCA show that the majority of the data (~ 82 %) could be explained by 2 principal components. The relationship between the sensory attributes and parameters from the different physical tests (rheology, texture analysis and force plate analysis) will be discussed separately.

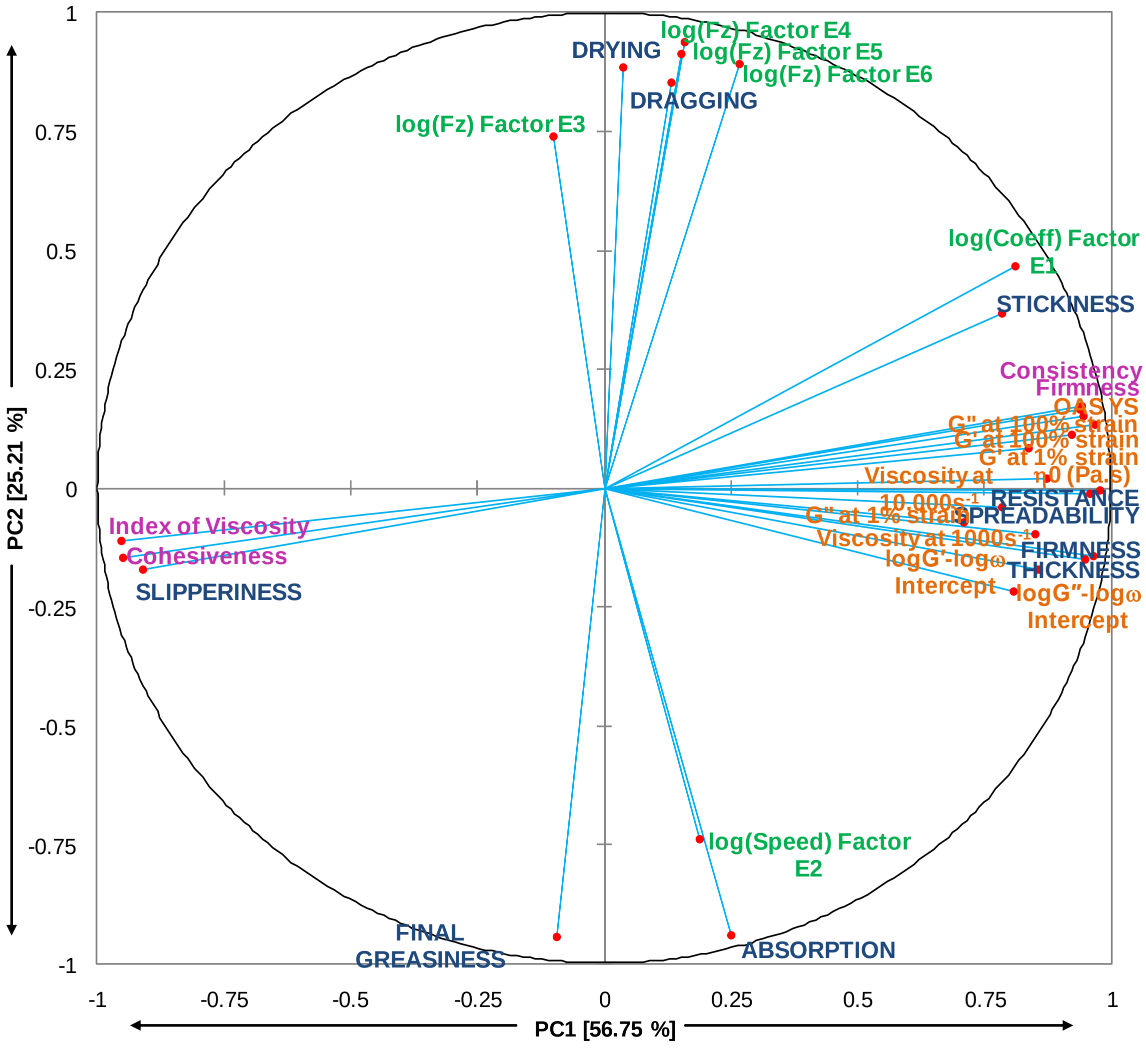


Figure 4.1: *PCA correlation circle showing the relationship between sensory attributes, and physical parameters derived from rheology, texture analysis and force plate analysis.*

4.1.1 Relationship between sensory attributes and rheological parameters

Rheological parameters were all highly correlated to PC1 (see Figure 4.1) and therefore with the sensory attributes on PC1, which were all related to initial application procedures (see explanation in Chapter 3.1.2 and attribute definitions in Chapter 2.3.2.3). The correlation of yield stress and zero shear viscosity values with initial skin cream application procedures is understandable, as such procedures relate to preliminary forces acting on the skin cream samples. Likewise values from frequency sweeps and the G′ at 1 % strain from amplitude sweep measurements were all taken before the sample had undergone irreversible deformation hence their relationship with initial skin cream application procedures.

It is interesting that the viscosities at 1000 s^{-1} and 10,000 s^{-1} and the G′ and G″ at 100 % strain were also correlated to PC1 as it might be expected that they would be related to secondary application procedures (see Chapter 3.1.2) since the product has undergone further deformation through high shear. However, the protocols by which attributes on PC2 were rated involved the samples being absorbed into the skin (to different extents) and therefore for these attributes the panel were measuring the interaction of the cream with the skin rather than the cream alone. All rheological measurements were carried out on fresh cream samples that did not absorb into anything or dry out (the peltier hood was used to prevent drying out) so during the course of the measurements they just deformed. This could explain why none of the rheological parameters were related to PC2.

Similar findings were reported by Parente et al. (2005) who studied the sensory properties of emollients used in cosmetics. They found that sensory attributes relating to mechanical instances of application, such as difficulty of spreading, stickiness and slipperiness were related to the viscosity of the products as measured using a Brookfield LVT viscometer (spindle number 1, 60 rpm, 20° C).

Brummer and Godersky (1999) found that the viscosity at the onset of flow was not related to primary skin feeling (sensations at the start of cream application) (see Chapter 1.8). Likewise in this research the complex viscosity and yield strain values at which the yield stress was taken were not related to attributes measured during initial interaction of cream with the skin.

Wang et al. (1999) found that rheological properties of creams had little effect on their moisturising efficacy as determined through TEWL and SC measurements (see Chapter 1.8). Likewise in this research it was found that the rheological properties had limited correlation to the sensory attributes related to absorption of the cream into the skin (attributes on PC2) i.e. attributes that influence the moisturising efficacy of the sample. The research carried out by Wang et al. (1999) also showed limited correlation of rheological results with sensory attributes measured by a panel (attributes were measured prior to and after absorption). They based their correlation on Steven's Equation:

$$S = P^{\alpha} \tag{4.4}$$

where S is the magnitude of a sensory attribute, P is the physical property and α, the magnitude of the exponent. Logged values for frequency sweep parameters (G' and G'' at 1 rad.s^{-1} as measured at 10 % strain which was within the LVD) were plotted against logged values for the sensory attributes. The value of the exponent (slope of the line) was < 0.1 for all parameter-attribute correlations suggesting limited correlation of frequency sweep parameters with sensory attributes. On the other hand, using data obtained in this research, therefore sensory QDA scores for attributes on PC1 and results from oscillation frequency sweep measurements, exponent values ranged between 0.5 to 0.6 (firmness, thickness, resistance and spreadability) with values of -0.2 and 0.1 for the attributes slipperiness and stickiness respectively. This indicates that in this case for some attributes a greater correlation between sensory attributes and rheological parameters exists, as was demonstrated through PCA (see Figure 4.1). Note that the G' and G'' at 1 rad.s^{-1} are not included in the PCA since they were similar to the G′ and G″ at 1 % strain from amplitude sweep measurements.

The differences between the results from the study by Wang et al. (1999) and this study could be related to the different cream samples used or the different sensory rating protocols, however the major difference between the studies is the fact that Wang et al. centrifuged the cream samples at 800 rpm for 5 minutes and left them to stand overnight prior to rheological measurements. The centrifugation may have affected the samples resulting in minimal correlation between sensory and rheological data. On the other hand in this study, cream samples were treated in the same manner for both sensory and physical measurements hence the greater correlation between rheological and sensory data.

4.1.2 Relationship between sensory attributes and back extrusion parameters

The back extrusion parameters were also all correlated to PC1. This makes sense as the back extrusion test measures the deformation experienced by the sample as the probe enters and is withdrawn from the sample (see Chapter 2.4.2). Likewise attributes on PC1 were all related to the sample undergoing initial deformation (see attribute protocols Table 2.3, Chapter 2.3.2.3). For example the firmness parameter measured during back extrusion could be crudely related to the trained panel's measurement of 'firmness', which involved gently dipping the index finger into the pot of cream and assessing how firm the sample was. In the case of back extrusion an aluminium probe (rather than a finger) was dipped into the pot of cream and removed whilst forces involved were recorded.

4.1.3 Relationship between sensory attributes and force plate parameters

It was hypothesised that force plate parameters may be related to secondary skin cream application procedures as they measure the frictional forces involved in spreading a sample of cream on synthetic skin (Bioskin) and the effects of sample drying on these values. Results showed that parameters extracted from force plate measurements (speed and load factors) were indeed related to PC2, although the log(Coefficient) factor (episode 1) was related to PC1, see Figure 4.1.

Sensory attributes on PC2 describe later stages of skin cream application including absorption of cream into the skin (see Chapter 3.1.2). The force plate parameters correlated to these attributes were mainly from later episodes in which the cream would be present as a thinner layer between surfaces and it would have been subjected to air drying for longer. Similar conditions applied to the creams when measured for sensory attributes on PC2, hence the correlation. The log(Coefficient) factor from episode 1 (initial stages of experiment) was found to be related to PC1 suggesting limited stroking is related to initial skin cream application procedures.

Sensory protocols used by the trained panel to measure the attributes on PC2 were given in Chapter 2.3.2.3, Table 2.3. Although these involved different time factors (20 seconds) to those used in force plate measurements (6 episodes each lasting 40 seconds spread out over 10 minutes), it is clear that the load applied will affect the sensory perception of these properties. Typically the higher the load, the higher the friction will be (see Chapter 1.7.1 and Figure 3.49, Chapter 3.5) this is likely to increase the drying or dragging feel of the cream on the skin and reduce the greasiness.

The final protocol used by the trained panel for rating absorption was given in Chapter 2.3.3.2. In effect panellists were rating the speed at which samples were absorbed within a 30 second application procedure. It is clear that the speed of stroking is likely to affect the absorption behaviour. In general, the faster the stroking, the faster the cream will be absorbed hence the correlation of absorption with the log(Speed) factor (r = 0.779).

As mentioned in Chapter 2.4.3.2, the log(Coefficient) factor provided an estimate of the overall frictional properties of the cream at typical loads (0.5 N) and speeds (100 $mm.s^{-1}$) involved in stroking. It might be expected that a stickier cream would have higher friction properties and therefore a higher log(Coefficient) factor which explains why this parameter was correlated to the stickiness (r = 0.820), see Figure 4.1.

Further understanding of physical parameters that provide good correlation with sensory properties can be gained by predictive modelling as discussed below in Chapter 4.2.

4.2 PREDICTIVE MODELLING

Physical parameters identified through PCA as being related to the sensory attributes (see Chapter 4.1) were used to create predictive models. Although PCA showed correlation of texture analysis (TA) and rheological data with sensory attributes on PC1 only (see Figure 4.1), predictive modelling was still carried out for all attributes for all parameters in case any relationships were overlooked in PCA. However, the predictive ability of models in which rheological and TA parameters were used to predict textural properties of attributes on PC2 was found to be poor, therefore these models are not discussed.

Separate models were created for the different physical tests (rheology, texture analysis and force plate analysis) as it would be more efficient if parameters extracted from one type of physical measurement could produce models to predict all the textural properties expressed by PC1 and PC2. These relationships are therefore discussed separately.

4.2.1 Predicting textural properties from rheological data

Preliminary models showed that all sensory attributes on PC1 could be explained by rheological parameters from oscillatory tests only (G′ and G″ at 100 % strain and logG′-logω and logG″-logω intercepts). Inclusion of steady shear parameters in these models did not enhance model performance therefore they were not included in the final models. Further modelling revealed that using amplitude sweep parameters only including a combination of average values and the logarithm of these values produced the most robust models.

Models for predicting textural sensory properties from rheological data are given in Table 4.2. Statistical values describing goodness of fit are also included. Predictive models in which sensory properties on PC1 were predicted from rheological parameters all showed high predicted and adjusted R^2 values with good agreement (difference < 0.2 between them). Final model equations show that all attributes on PC1 could be predicted by just 4 parameters: G′ at 100 % strain, logG″ at 100 % strain, logG′ at 1 % strain and logG″ at 1 % strain. Firmness, thickness, resistance and spreadability models all included the same model terms with different factors by which they should be multiplied.

Table 4.2: *Predictive model equations and goodness of fit data for predicting sensory properties from rheological data.*

Attribute	Final model equations	R^2	Adjusted R^2	Predicted R^2
PC1				
FIRMNESS	Firmness = -2.834 $+1.161 \times 10^{-3}$ * G′ at 100 % strain +3.299 * logG″ at 100 % strain	0.960	0.951	0.910
THICKNESS	Thickness = -2.342 $+8.793 \times 10^{-4}$ * G′ at 100 % strain +3.322 * logG″ at 100 % strain	0.949	0.938	0.909
RESISTANCE	Resistance = -1.997 $+2.375 \times 10^{-3}$ * G′ at 100 % strain +2.130 * logG″ at 100 % strain	0.934	0.919	0.860
SPREAD-ABILITY	Spreadability = -1.121 $+2.746 \times 10^{-3}$ * G′ at 100%strain +1.377 * logG″ at 100% strain	0.908	0.888	0.859
STICKINESS	Stickiness = -9.181 +13.201 * logG′ at 1 % strain -10.696 * logG″ at 1 % strain	0.808	0.766	0.634
SLIPPERINESS	Slipperiness = +8.186 -3.326×10^{-3} * G′ at 100 % strain	0.823	0.806	0.770

The parameters involved in the models relate to elastic (G′) and viscous (G″) properties of the creams at high (100 % strain) and low (1 % strain) deformation. High values for G′ and G″ at 100 % strain were associated with creams that were thicker (see Chapter 3.3.1). At 1 % strain the samples were within the LVD therefore elastic behaviour was dominating (G′ > G″, tanδ < 1) indicating that the samples were showing complete reversible deformation behaviour (see Chapter 1.5.1). On the contrary, at 100 % strain the majority of samples had undergone some irreversible deformation due to the greater strain applied and viscous behaviour was dominating (G″ > G′) or closer to dominating (tanδ values closer to or greater than 1) (see Chapter 3.3.1, Figure 3.32). If a sample shows reversible deformation behaviour after a high strain has been applied (i.e. G′ > G″ at 100 % strain), this provides a more accurate measurement of the thickness (and associated properties) than its properties at low strain deformation.

Overall the higher the G′ and logG″ at 100 % strain values, the higher the firmness, thickness, resistance and difficulty of spreading (positive side of PC1) and the lower the slipperiness (negative side of PC1). This is understandable as all these attributes are related to the thickness of the cream sample. A thicker sample is likely to be less slippery due to the thickness of the sample creating drag and therefore more resistance (increase in resistance) this in turn accounts for the greater difficulty of spreading and the resulting firmer, thicker sample (as was observed in results).

The stickiness model showed that the higher the elasticity at low deformation (logG′ at 1 % strain), the greater the stickiness and the more viscous properties present at low deformation (logG″ at 1 % strain), the lower the stickiness (see Table 4.2). The elastic behaviour of the sample appears to be important in determining the overall stickiness. A sample with high elasticity is likely to be stickier. Note that parameters included in the stickiness model did not have the best correlation with the attribute stickiness (compared to the other parameters) but in combination they produced the best model and were therefore included in Table 4.2.

4.2.1.1 Model validation

Results from the analysis of the 40 model creams were used to check the robustness of these models since data for all rheological parameters had been obtained for the 40 creams. Figure 4.2 shows the predicted versus actual data for the attributes firmness, slipperiness and stickiness respectively (plots for thickness, resistance and spreadability are not shown as these models are similar to the model for firmness). The figures include x = y lines to allow for visualisation of residual error (the closer the data points are to the line, the better the fit).

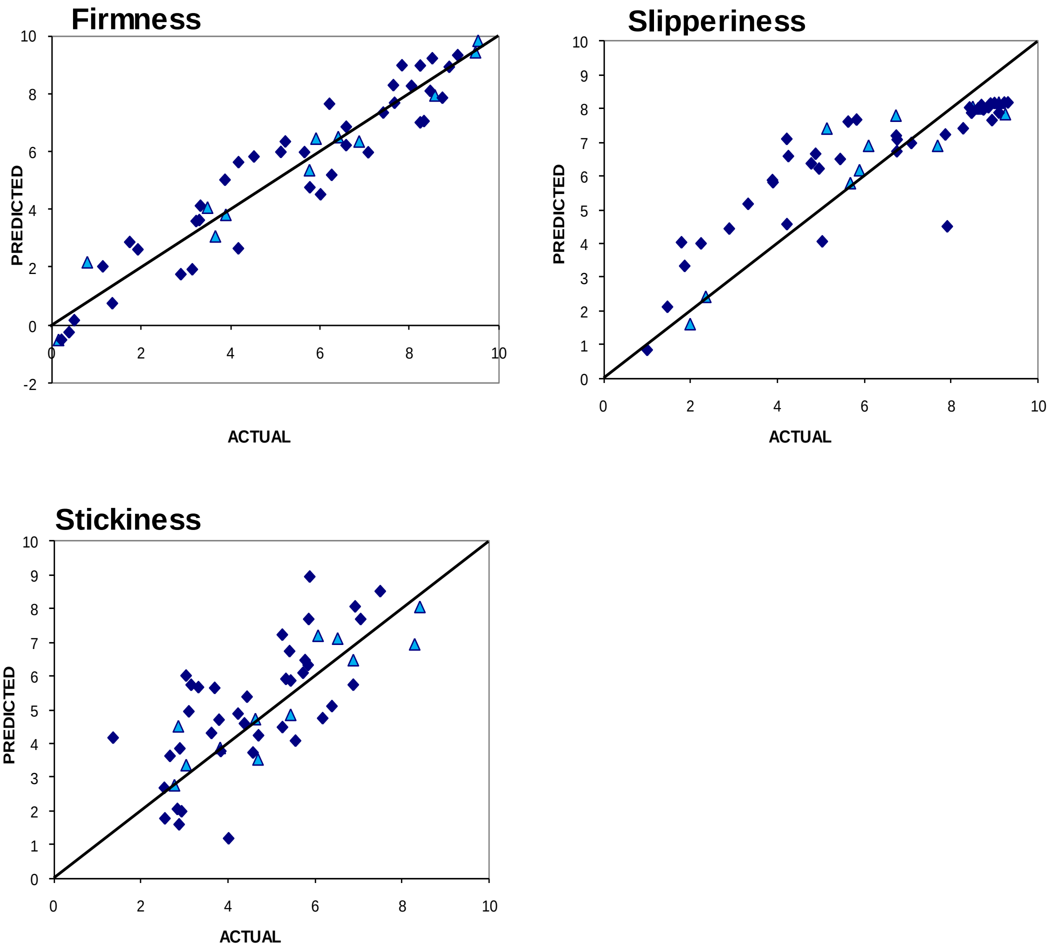


Figure 4.2: *Predicted versus actual scores for the attributes firmness, slipperiness and stickiness where the results for the consumer study creams (▲ data points) are compared with the 40 model skin creams (◆ data points). The x = y line is also included to aid visualisation of residual error.*

Data points in the firmness validation plot (Figure 4.2) follow the x = y line well suggesting good predictive ability of the model. Similar model relationships were found for the attributes thickness, resistance and spreadability suggesting that these models can be used to make predictions about skin creams (R^2 = 0.9). The slipperiness and stickiness models show slightly weaker trends whereby data points are further away from the x = y lines however the R^2 values were reasonable (R^2 = 0.8) which suggests that they could be used to make predictions. On the other hand the predicted and adjusted R^2 values (in particular for the stickiness model) are slightly lower suggesting a weaker predictive ability. Therefore the stickiness and slipperiness models should not be used to make significant predictions. However, if a general idea of the stickiness or slipperiness was required then these models could be used as they explain ~ 80 % of the variation in the slipperiness or stickiness characteristics of the cream samples. Demartine and Cussler (1975) also found that predicting the stickiness of skin creams was less successful than other attributes e.g. spreadability (see Chapter 1.8). This suggests that stickiness is a more complex attribute regarding sensory perception.

4.2.2 Predicting textural properties from texture analysis data

Predictive models obtained for predicting sensory properties from texture analysis data are given in Table 4.3. Note that negative values for cohesiveness and index of viscosity were used. Once again all equations were linear and there were no interactions between model terms. It is clear that these models are not as robust as those obtained using rheological parameters to predict sensory properties (compare R^2 values in Table 4.3 with Table 4.2). Predicted and adjusted R^2 values were in reasonable agreement (difference < 0.2 between them) suggesting good model fit. However, in some cases the R^2 values were quite low in particular for the attributes firmness, thickness and stickiness. For all models predicting sensory properties from texture analysis results, only one parameter was involved in each model, either the cohesiveness or the consistency.

It was thought that the TA parameter firmness may be involved in the predictive model for sensory firmness due to the correlation found between the results (see Chapter 3.4). Preliminary models showed that the sensory firmness could be predicted from the TA firmness alone, although the R^2 values were slightly lower (R^2 = 0.701, adjusted R^2 = 0.673, predicted R^2 = 0.574). Therefore only the most robust models are presented in Table 4.3.

Table 4.3: *Predictive model equations and goodness of fit data for predicting sensory scores from texture analysis.*

Attribute	Final model equations	R^2	Adjusted R^2	Predicted R^2
FIRMNESS	Firmness = +3.078 -3.643×10^{-3} * TA cohesiveness	0.722	0.694	0.574
THICKNESS	Thickness = +3.625 -3.403×10^{-3} * TA cohesiveness	0.684	0.652	0.526
RESISTANCE	Resistance = +1.750 -3.831×10^{-3} * TA cohesiveness	0.835	0.818	0.754
SPREAD-ABILITY	Spreadability = +1.279 -3.532×10^{-3} * TA cohesiveness	0.836	0.820	0.769
STICKINESS	Stickiness = +3.937 $+1.632 \times 10^{-4}$ * TA consistency	0.633	0.596	0.404
SLIPPERINESS	Slipperiness = +8.356 $+3.068 \times 10^{-3}$ * TA cohesiveness	0.829	0.812	0.759

The texture analysis parameter cohesiveness is a measure of how strong the internal bonds making up the body of the product are. The **more negative** the values, **the greater the cohesiveness** of the sample (see Chapter 2.4.2). The predictive models have shown that high values for cohesiveness are related to a low firmness, thickness, resistance and difficulty of spreading and a high slipperiness. This makes sense as high values for TA cohesiveness (more positive) relate to samples that are not as cohesive. Therefore the bonds between molecules making up the skin creams are weaker resulting in products that are less firm, less thick, have lower resistance, are easier to spread and are more slippery. The TA parameter consistency was related to the stickiness where high values for consistency represent samples with a high stickiness. The R^2 values for this attribute were also low suggesting limited accuracy in the model so it was decided this model should not be used to make predictions.

Figures 4.3 and 4.4 provide predicted versus actual plots for the attributes thickness, spreadability, stickiness and slipperiness (firmness and resistance data are not shown due to the similarity between thickness and spreadability results respectively). The attributes spreadability, resistance and slipperiness showed reasonable correlation between predicted and actual values (R^2 = 0.8) whereas relationships for the thickness, firmness and stickiness were not ideal (poor correlation with the x = y lines and low R^2 values, R^2 = 0.6 – 0.7).

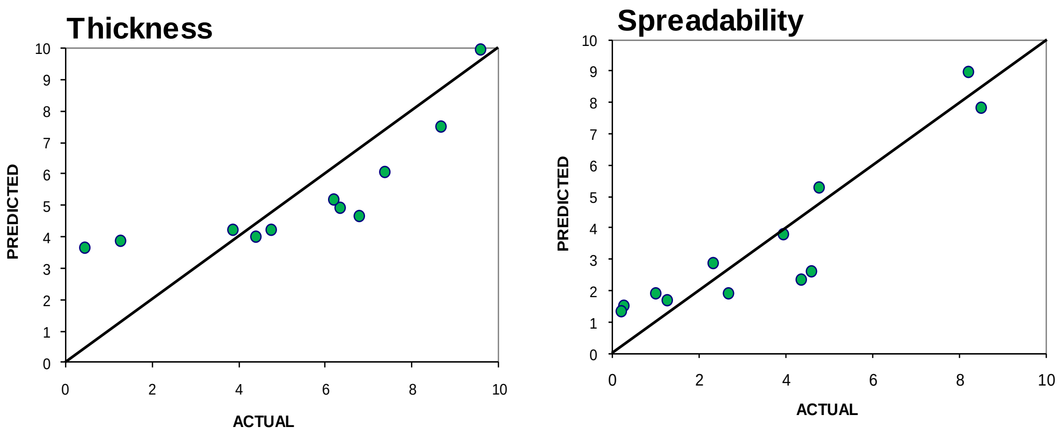


Figure 4.3: *Predicted versus actual scores for the attributes thickness and spreadability for the 12 consumer study creams. Plots include x = y lines.*

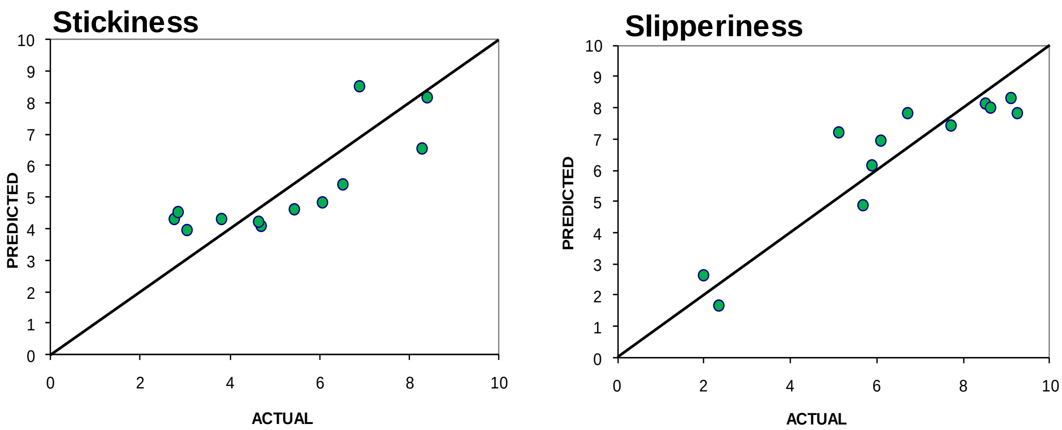


Figure 4.4: *Predicted versus actual scores for the attributes stickiness, and slipperiness for the 12 consumer study creams. Plots include x = y lines.*

4.2.3 Predicting textural properties from force plate data

Predictive models as derived from force plate parameters are given in Table 4.4. Most equations were linear except for the drying and dragging models, which included $\log_{10}$ transformations. There were no interactions between model terms in the predictive equations. Models showed good agreement between predicted and adjusted R-squared values (difference < 0.2 between them) suggesting good model fit. However, as found with the TA data, in some cases the R^2 values were low in particular for the attribute spreadability.

The log(Coefficient) factor is a measure of the overall frictional properties of the creams at 100 $mm.s^{-1}$ and 0.5 N. High values for the log(Coefficient) factor (episode 1) related to a high firmness, thickness, resistance, spreadability and stickiness (positive side of PC1) and a low slipperiness (negative side of PC1), see Figure 4.1, Chapter 4.1. This is understandable as during episode 1, the cream samples were in their freshest form, before a high level of stroking deformation hence they were in a thicker, less slippery state.

The equations in Table 4.4 show that the higher the log(Fz) factor (episode 6), the lower the firmness, thickness, resistance and stickiness and the higher the dragging properties of the cream. In Chapter 3.5, Figure 3.50, the friction coefficient is plotted against the load, the slope of this graph is related to the log(Fz) factor, see Chapter 2.4.3.2. Results revealed that a negative slope for friction coefficient against Fz suggested that the samples were more lubricating. Therefore higher values for the log(Fz) factor relate to samples that are less lubricating on increase in load. Poor lubrication is generally associated with thinner samples therefore samples that are less firm, less thick, have lower resistance and in this case are less sticky and more dragging (see equations in Table 4.4).

Table 4.4: *Predictive model equations and goodness of fit data for predicting sensory scores from force plate analysis.*

Attribute	Final model equations	R^2	Adjusted R^2	Predicted R^2
PC1				
FIRMNESS	Firmness = +8.599 -20.101 * log(Fz) factor E6 +30.518 * log(Coeff) E1	0.863	0.832	0.721
THICKNESS	Thickness = +8.851 -19.488 * log(Fz) factor E6 +29.330 * log(Coeff) E1	0.864	0.834	0.742
RESISTANCE	Resistance = +7.463 -14.196 * log(Fz) factor E6 +27.442 * log(Coeff) E1	0.780	0.731	0.507
SPREAD-ABILITY	Spreadability = +6.390 +16.642 * log(Coeff) E1	0.566	0.523	0.407
STICKINESS	Stickiness = +8.134 -23.292 * log(Fz) factor E6 +20.504 * log(Fz) factor E4 +17.261 * log(Coeff) E1	0.835	0.774	0.575
SLIPPERINESS	Slipperiness = +3.695 -15.729 * log(Coeff) E1	0.664	0.630	0.527
PC2				
DRYING	Log(Drying) = +0.808 +4.194 * log(Fz) factor E4	0.784	0.762	0.695
DRAGGING	Log(Dragging) = +0.719 +2.999 * log(Fz) factor E6	0.716	0.686	0.557
FINAL GREASINESS	Final Greasiness = +1.403 -24.243 * log(Fz) factor E4	0.760	0.736	0.664
ABSORPTION	Absorption = +4.680 -10.955 * log(Fz) factor E4 +9.176 * log(Speed) factor E2	0.796	0.752	0.646

High values for the log(Fz) factor for episode 4 related to creams with a high drying nature and a low final greasiness and absorption[1] (faster absorption). This is understandable as already mentioned, high values for the log(Fz) factor relate to less lubricating samples. If a sample provides poor lubrication it is likely to be a cream with high drying properties, rapid absorption and therefore low final greasiness. On the other hand for the attribute stickiness, the predictive equation shows that the higher the log(Fz) factor (episode 4), the higher the stickiness whereas higher values for the log(Fz) factor (episode 6) relate to a lower stickiness. It can only be assumed that this is due to the episodes involved. During episode 4, sample thickness will be higher (having undergone less stroking deformation) and therefore possibly stickier. This, however, is only an assumption and will vary with cream.

The absorption equation in Table 4.4 shows that high values for the log(Speed) factor episode 2 relate to higher values for absorption (slower absorption rate). High values for log(Speed) factor also relate to less lubricating samples (see Figure 3.52, Chapter 3.5). In previous discussion it was assumed that less lubricating samples absorb quicker, but in this case the higher the log(Speed) factor, the slower the absorption. However, it should be noted that the episode at which the log(Speed) factor affects absorption is episode 2, so in this episode layers of cream will still be relatively thick. A sample may be less lubricating in higher episodes (when thinner) but more lubricating in lower episodes i.e. episode 2 (when thicker) thus creating a slower absorption rate.

Ultimately, it is apparent that the speed and loads applied during application of skin cream are likely to affect the absorption rate, which is why these parameters are in the model equation for absorption. The reason why the different episodes are

[1] Note when rating the consumer study creams, the scale ends for rating absorption went from 'fast to slow', see Chapter 2.3.3.2 whereas previously the rating scale went from slow to fast (rating 40 model creams). Therefore in these models, low values for absorption relate to fast absorption and high values relate to slow absorption.

included is because the properties of the creams at different stages of drying also affect the absorption. Thicker samples will absorb at a slower rate hence the lower episode (log(Speed) factor E2) was related to slower absorption, whereas later episodes (log(Fz) factor E4) were related to faster absorption.

Predicted versus actual plots for most of the models are given in Figures 4.5 - 4.8. Thickness and resistance plots are not shown due to their similarity with the firmness plot. Models for the attributes firmness and thickness were good as can be seen by the spread of data on the x = y line (Figure 4.5) and the high R^2 values (R^2 = 0.86, Table 4.4). Weaker relationships were observed for other attributes on PC1 as can be seen by the lower R^2 values (spreadability and slipperiness) and the greater distance between predicted and adjusted R^2 values (resistance and stickiness). Models for attributes on PC2 were also weaker which was surprising as PCA results suggested that a closer relationship between force plate parameters and attributes on PC2 exists than that between force plate parameters and attributes on PC1 (see Figure 4.1). As with TA models, validation could not be carried out since force plate measurements were only carried out on the 12 consumer study creams that were used to create the predictive models.

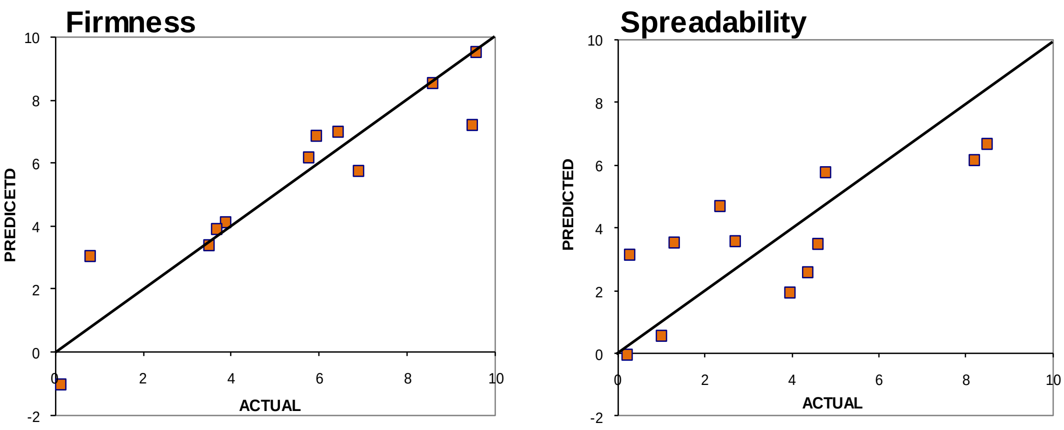


Figure 4.5: *Predicted versus actual for attributes firmness and spreadability for the 12 consumer study creams (x = y lines are also shown).*

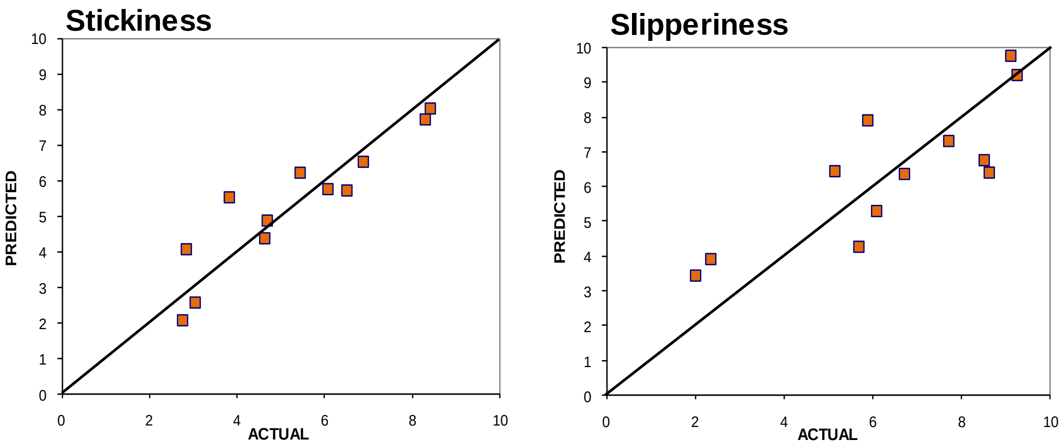


Figure 4.6: *Predicted versus actual for attributes stickiness and slipperiness for the 12 consumer study creams (x = y lines are also shown).*

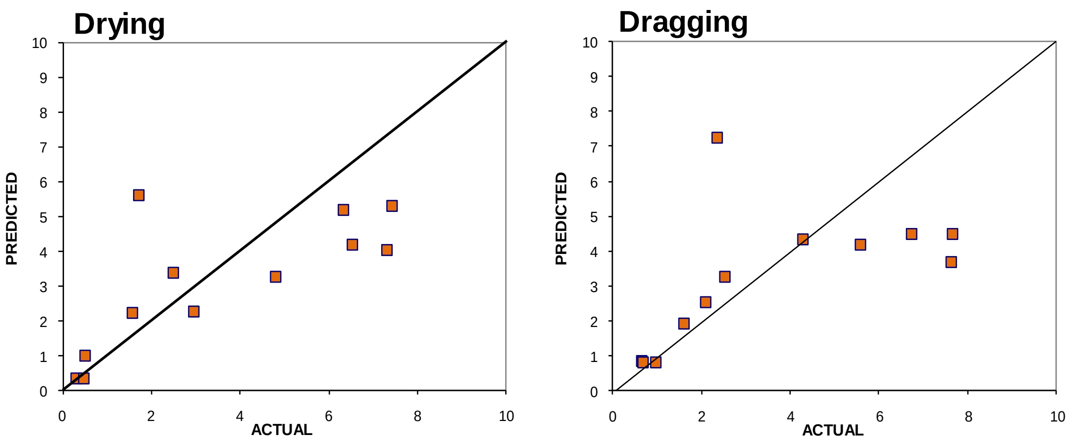


Figure 4.7: *Predicted versus actual for attributes drying and dragging for the 12 consumer study creams (x = y lines are also shown).*

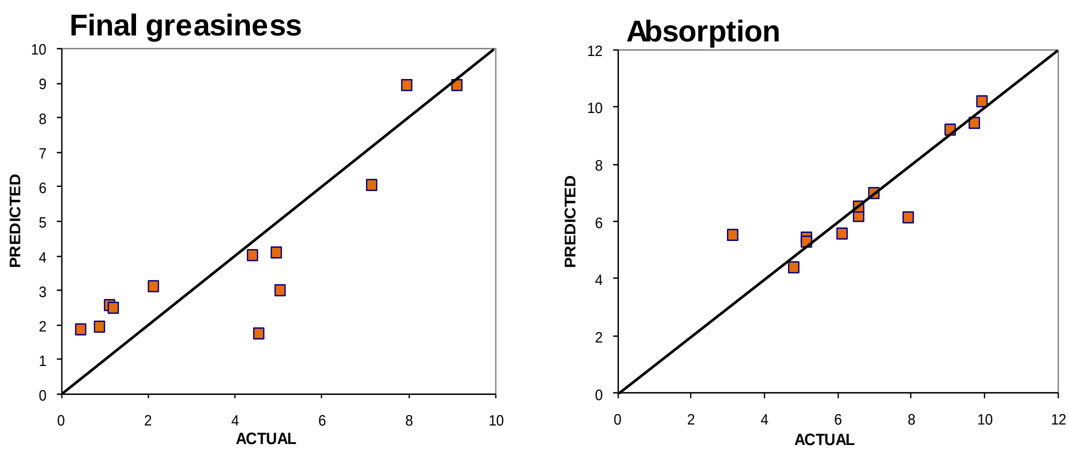


Figure 4.8: *Predicted versus actual for attributes final greasiness and absorption for the 12 consumer study creams (x = y lines are also shown).*

4.2.4 All inclusive models

Models can be produced including a combination of rheology, force plate and texture analysis model terms. Such models were generated for predicting attribute properties on PC2. These models had higher R^2 values and good agreement between predicted and adjusted R^2 values for all attributes therefore suggesting greater predictive ability than models including parameters from one physical test only. The most robust models are given in Table A12.1, Appendix XII. These models have not been discussed, as they have not been validated so their predictive ability is uncertain. Also it was thought that creating models using parameters from one type of physical measurement would be more beneficial as it would save both analysis and measurement time. These models include parameters from two of the physical tests therefore practically they are not very efficient for predicting sensory properties.

4.2.5 Summary

Overall it was found that oscillation amplitude sweep parameters, G′ and logG″ at 100 % strain, and the logG′ and logG″ at 1 % strain provided the best correlations with the majority of the attributes on PC1 and can therefore be used in predictive modelling for these attributes. However, the stickiness model showed weaker predictive ability and should therefore be approached with caution. TA results were also correlated to PC1 attributes although the predictive models were not as robust as those produced from rheological data. Force plate data enabled models explaining attributes on PC2 to be produced although the resulting models were not that highly predictive (R^2 = 0.7) and further validation is required to be assured of their accuracy in making predictions.

4.3 RELEVANCE OF PREDICTIVE MODELS TO CONSUMER LIKING

Knowledge of product attributes liked by consumers provides additional value to the predictive models i.e. only models predicting attributes relevant to consumer liking are beneficial and worth pursuing. The consumer study revealed that the firmness and thickness of creams was very important regarding consumer satisfaction, samples that were too thin or too thick were less popular. Resistance and spreadability properties were also important in terms of how easy it was to apply the sample to the skin and the attributes on PC2 (in particular the drying, dragging and final greasiness) were important regarding the performance of the cream (see Chapter 3.2.7).

The firmness model including rheological parameters was the most robust of all models created (R^2 = 0.960) therefore it will be used as an example here. If a cream has been produced by a manufacturer and the company are unsure whether to pursue it as a new product then the oscillation amplitude sweep described in Chapter 2.4.1.1.1 can be carried out and results used to calculate the G′ at 100 % strain and the logG″ at 100 % strain. The values for these parameters can then be used to calculate the predicted firmness using the firmness equation (see Table 4.2, Chapter 4.2.1). For example if the values for G′ at 100 % strain and logG″ at 100 % strain were 438 and 2.31 respectively then the sample would have a predicted firmness of 5.29:

Predicted firmness

$$\mathbf{= -2.834 + 1.161 \times 10^{-3}\ (G' \text{ at } 100\ \%\ \text{strain}) + 3.299\ (\log G'' \text{ at } 100\ \%\ \text{strain})}$$

$$= -2.834 + 1.161 \times 10^{-3}\ (438) + 3.299\ (2.31)$$

$$= \mathbf{5.29} \qquad (4.5).$$

It is likely that the consumer would like this sample since it has a medium firmness value and is therefore not too firm or too thick. However as discussed in Chapter 3.2, different types of consumer had different preferences therefore the attributes affecting consumer liking and the limits for parameters that predict such attributes are summarised in Table 4.5. Looking at Table 4.5 reveals that this firmness value was liked by ~ 54 % of consumers that participated in the consumer study (consumers with vector models in cluster 2 and ideal point models). It is not possible to please all consumers however, comparing the attribute limits for creams liked by consumers with different model types it is possible to determine the attribute properties that would be liked by the majority of consumers. An ideal firmness would be between 3.5 and 4 since this would be liked by ~ 67 % of consumers capturing 3 consumer groups (consumers with vector models in clusters 2 and 3 and ideal point models). The limits in Table 4.5 are therefore useful to use alongside predictive equations to adjust formulations so creams catering for the largest group of consumers could be manufactured. This example looked at the attribute firmness only, however in reality the properties of other attributes should be considered too as a combination of different attributes affect consumer liking (see Table 4.5 and Chapter 3.2.3).

The PCA correlation circle showed that attributes on PC1 (excluding stickiness) were highly correlated in particular the firmness and thickness ($r = 0.997$) and the resistance and spreadability ($r = 0.975$). Likewise attributes on PC2 were highly correlated in particular drying and dragging ($r = 0.987$) and the final greasiness with drying and dragging ($r = 0.929 – 0.956$). Therefore if time limitations are a problem, the most efficient usage of predictive models would be to take one model predicting each of the highly correlated attributes. In terms of predictive ability, the best models to use would be the firmness and resistance models including oscillation amplitude sweep parameters, which have proven to be robust enough for making predictions (see Chapter 4.2.1, Table 4.2 and Figure 4.2). The drying model in terms of force plate parameters showed the greatest predictive ability for attributes on PC2. This model however is much weaker ($R^2 = 0.784$) than the firmness (R^2 = 0.960) and resistance (R^2 = 0.919) models and it has not been validated therefore it would not be suitable for making important predictions.

Table 4.5: *Sensory attribute and instrumental parameters levels affecting consumer liking of model skin creams*

Consumer Group	**Attribute levels liked by > 50 % consumers**	**Instrumental parameter limits liked by > 50 % consumers**
Vector model consumers cluster 1 (28.2 % participants)	**Drying < 3** **Dragging < 3** **Final greasiness > 3** **Absorption > 6.5**	**log(Fz) factor E6: -0.269 to -0.106** **log(Fz) factor E4: -0.311 to -0.067** **log(Speed) factor E2: 0.072 to 0.233**
Vector model consumers cluster 2 (30.3 % participants)	**Firmness < 8** **Thickness < 7** **Resistance < 6** **Spreadability < 5** **Slipperiness > 6**	**G′ at 100 % strain: 5 to 609 Pa** **G″ at 100 % strain: 5 to 491 Pa**
Vector model consumers cluster 3 (14.1 % participants)	**Firmness < 4** **Thickness < 5** **Resistance < 4** **Spreadability < 3** **Slipperiness > 6.5**	**G′ at 100 % strain: 5 to 110 Pa** **G″ at 100 % strain: 5 to 114 Pa**
	Stickiness < 5	**G′ at 1 % strain: 38 to 1217 Pa** **G″ at 100 % strain: 5 to 95 Pa**
	Final greasiness < 5 *Absorption 3 - 7*	*log(Fz) factor E4: -0.111 to -0.014* *log(Speed) factor E2: -0.110 to 0.142*
Ideal point model consumers (23.2 % participants)	**Firmness 3.5 – 7** **Thickness 4 - 8** **Resistance 1 - 6** **Spreadability 1 - 5** **Slipperiness 5 - 9**	**G′ at 100 % strain: 57 to 609 Pa** **G″ at 100 % strain: 59 to 491 Pa**
	Drying 1.6 – 6.3 *Dragging 1.6 – 5.3* *Absorption 5 - 7*	*log(Fz) factor E4: -0.111 to -0.014* *log(Fz) factor E6: -0.145 to 0.047* *log(Speed) factor E2: -0.110 to 0.142*
Note that attributes and parameters in **bold** have the main effects on consumer liking whereas attributes in *italics* play a less dominant role in consumer liking (see Chapters 3.2.4 – 3.2.7).		

4.3.1 Summary

Overall the ability to predict the sensory properties of creams from instrumental parameters is useful, however understanding which of these properties are key to consumer acceptability is vital for optimum use of the models. These models could be used in product development to determine whether products should be taken to market or not. In particular the firmness and resistance models would be useful, as extreme samples in terms of these attributes are undesirable from a consumer's perspective. The drying or final greasiness models would also be useful if they had been validated.

4.4 CONCLUSION

The relationship between physical parameters and sensory properties of skin creams was visualised using PCA. Predictive models were then created using physical parameters related to PC1 and PC2. These models allow future predictions about the sensory properties of creams to be made from physical data thus eliminating the expensive, time consuming sensory analysis stages. Rheological parameters best described attributes on PC1 while force plate data could be used to make predictions about attributes on PC2. Models describing sensory attributes on PC2 have not been validated so their use to make predictions should not be done in total confidence. The ability of models to predict attributes important to consumers was also highlighted in terms of their application on a wider scale in product development.

5. OVERALL CONCLUSIONS AND FUTURE WORK

The main aims of this PhD were to understand the relationship between sensory attributes and rheological parameters of skin creams and to understand which product attributes are key drivers of consumer acceptability (see Chapter 1.2). Models skin creams (40 samples, see Table 2.2, Chapter 2.2.1) with a wide range of textural properties formed the basis of this research. Rheological and sensory properties were measured and correlated through PCA and predictive modelling. Texture and force plate analysis were also carried out to complement the rheological measurements providing additional information about the model cream characteristics. A subset of 12 model skin creams was included in a consumer study that enabled hedonic data about the creams to be gained. Textural attributes liked by consumers were identified through cluster analysis and external preference mapping. Results were used to add value to predictive models since models predicting attributes liked by consumers are practically more useful than models predicting attributes of no consumer relevance.

5.1 KEY FINDINGS

5.1.1 Relationship between sensory attributes and physical parameters

Sensory attributes measured during QDA by a trained sensory panel related to both initial application procedures (firmness, thickness, resistance, spreadability, stickiness and slipperiness) and secondary application procedures involving a time factor and different extents of absorption into the skin (drying, dragging, absorption and final greasiness). The PCA correlation circle showed that the initial application procedures were correlated to PC1 and secondary application procedures were correlated to PC2 (see Chapter 3.1.2).

Rheological and texture analysis parameters were all correlated to PC1 and therefore related to the attributes expressed by initial application procedures. These measurements were carried out on fresh cream samples that did not absorb into anything, hence the similarity with attributes such as firmness and thickness for which rating protocols involved limited interaction of cream with the skin. On the other hand force plate parameters were found to correlate mainly with attributes on PC2, which related to secondary application procedures (see Chapter 4.1). Force plate parameters quantify the frictional properties of cream under various states of drying, hence the correlation to attributes such as absorption and final greasiness, which involved absorption of the creams into the skin.

5.1.2 Understanding key drivers of consumer acceptability

Model skin creams were presented to consumers who rated how much they liked/disliked the feel of the samples on a LAM scale (where 0 = greatest imaginable dislike and 100 = greatest imaginable like) (see Chapter 2.3.3.4). Results revealed different groups of consumers for which different attributes were important but overall it was revealed that extreme samples were generally disliked or liked less by consumers in particular for the attributes firmness and thickness. In the case of resistance and spreadability, lower values were desired i.e. the upper extreme was disliked (creams that were too difficult to spread or had too high a resistance). For the attributes on PC2 it appeared that consumers preferred samples that were not too drying or dragging and therefore samples that added moisture to the skin (high final greasiness was preferable to low final greasiness) (see Chapters 3.2.4 – 3.2.7).

5.1.3 Ability to predict sensory properties of creams from physical parameters

Predictive models were developed that allowed sensory attributes to be predicted from physical parameters. Models including rheological parameters from oscillatory measurements (G′ at 100 % strain, logG″ at 100 % strain, logG′ at 1 % strain and logG″ at 1 % strain) provided the highest predictive ability (R^2 = 0.808 –

0.960) and validation of these models confirmed their ability to make reasonable predictions for attributes on PC1 (see Chapter 4.2.1). Weaker models were obtained for texture analysis and force plate parameters (see Chapters 4.2.2 – 4.2.3). Models to predict attributes on PC2 were created using force plate parameters. The predictive ability of these models was low (R^2 = 0.714 – 0.796) and model validation could not be carried out therefore they should not be used to make significant predictions.

5.1.4 Predictive models: a wider context

Models predicting attributes that were found to affect consumer liking (e.g. firmness and thickness) could play a significant role during new product development (see Chapter 4.3). Creams could be produced on a small scale and then measured rheologically and/or via force plate analysis, model equations could then be used to predict firmness and other attributes relevant to the consumer. This would provide an idea of how much the consumer would like the resulting creams indicating whether it is worth developing the cream further or not.

Creams used in this project were model skin creams containing no perfume or colour which are likely to affect consumer perception, therefore results are more likely to apply to creams on the market with limited colour or fragrance, in particular creams developed for consumers with sensitive skin e.g. eczema or cheaper own-label brands.

5.2 FUTURE WORK

This PhD has provided valuable information about the characteristics of creams relevant to consumer liking that can be measured by both sensory and physical measurements. It is clear that understanding why creams possess certain characteristics, e.g. why they are firm or why they have a high final greasiness, would be beneficial. These questions could be answered through investigating the microstructure of the creams which could be achieved through polarized light microscopy to obtain photomicrographs of the cream samples (Pena et al., 1994). Characterisation of the phase volume fraction and droplet size distribution of the cream samples would provide further information of the microstructure (Wibowo and Ng, 2001).

In addition to work understanding the microstructure of creams, further information about properties liked or disliked by consumers would be beneficial. The consumer study involved in this PhD looked at how much consumers liked or disliked the feel of cream samples as they applied them to the skin. It is however possible that other sensory factors including the appearance and smell of the samples may have affected consumer judgement (focus groups on consumer study creams showed the importance of appearance in consumer judgement in particular brand imaging). Therefore, another study investigating in more detail which properties consumers like and why, would add to this project. Such information could be gained through focus groups in which the model creams could be discussed.

Predictive models produced to describe sensory attributes in terms of physical parameters were reliable for attributes on PC1, however it was clear that models predicting attributes on PC2 could be improved with further research. Further investigation into physical parameters that could describe attributes on PC2 would be beneficial to allow more robust predictions about attributes on PC2 to be made, validation of these models would be crucial to ensure predictive ability is sound and to improve overall confidence in the models. However, the multifactorial nature of attributes liked by consumer's means that it can be difficult to find an instrument that can measure all attributes like a human assessor (Manuskiatti et al., 1998). Human perceptions of consumer products are the results of complex sensory and interpretation processes (Lawless and Heymann, 1998). For example, in contrast to an instrument, human soft tissue can deform during a test thus the mechanical stimulus perceived by a panellist or consumer will be different to that measured by an instrument. Also, humans subconsciously alter the way they assess products depending on the properties of the material they are sampling. For example if a cream is very thick, the consumer or panellist may apply more force to the sample to aid absorption of the cream into the skin. An instrument on the other hand is programmed to measure all samples in the same way hence the difficulty in finding an instrument that can accurately measure the attribute properties perceived by a human.

In summary, future work investigating the microstructure of creams, properties affecting consumer liking and instrumental measurements from which parameters predicting sensory properties can be gained, is suggested.

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APPENDIX

Appendix I

Table A1.1: *Properties of commercial skin cream samples used in focus group*

NAME OF CREAM	COMPANY	INGREDIENTS	ANY SPECIAL CLAIMS (including description of cream)	PROPERTIES (perfumed/ unperfumed, viscosity, colour)	PACKAG-ING	COST
DERMATO-LOGICAL E45 Moisturising Lotion	Crookes Healthcare Limited, Nottingham, NG2 3AA, UK	Aqua, Petrolatum, Isopropyl Palmitate, Paraffinum Liquidum, Glyceryl Stearate, Ceteth-20, Hypoallergenic Anhydrous Lanolin (Medilan^{R}), Phenoxyethanol, Methylparaben, Hydroxyethylcellulose, Carbomer, Propylparaben, Sodium Hydroxide, BHT.	Hypoallergenic. Soothes, softens & relieves dry and sensitive skin. For hands, body and face. Allergy screened – non-greasy. Dermatalogically tested	Unperfumed, white -opaque, quite runny – easy to pump!	Translucent, white package, 500mL pump (allows light through so can just see level of cream)	£3.49
Atrixo enriched moisturising cream	BDF Beiersdorf UK Ltd., Birmingham, B37 7YS. Beiersdorf Ireland Ltd., Santry, Dublin 9. Beiersdorf AG Hamburg	Aqua (water), Cetyl Alcohol, Myristyl Alcohol, Glycerin, Sorbitan Stearate, Isopropyl Palmitate, Ceteareth-3, Cera Microcristallina, Octyldodecanol, Dimethicone, Triceteareth-4 Phosphate, Paraffinum Liquidum, Panthenol, Phenoxyethanol, Sodium Carbomer, Methylparaben, Glycine Soja, Bisabolol, Butylparaben, Ethylparaben, Isobutylparaben, Propylparaben, Chamomilla Recutita, Parfum (Fragrance), Alpha-Isomethyl Ionone, Hexyl Cinnamal, Hydroxyisohexyl 3-Cyclohexene Carboxaldehyde, Benzyl Salicylate, Linalool, Citronellol, Geraniol, Hydroxycitronellal, Eugenol	Soothes and softens your hands, with Camomile	Perfumed, White – opaque, less runny than E45	200mL tub, yellow, opaque package, green lid.	£2.98

NIVEA soft Intensive Moisturising Cream for invigorated, smooth skin.	BDF Beiersdorf UK Ltd., Birmingham, B37 7YS. Beiersdorf Ireland Ltd., Santry, Dublin 9. Beiersdorf AG Hamburg.	Aqua (water), Paraffinum Liquidum, Myristyl Alcohol, Glycerin, Butylene Glycol, Alcohol Denat., Stearic Acid, Myristyl Myristate, Cera Microcristallina, Glyceryl Stearate, Hydrogenated Coco-Glycerides, Dimethicone, Simmondsia Chinensis, Tocopheryl Acetate, Polyglyceryl-2 Caprate, Sodium Carbomer, Phenoxyethanol, Lanolin Alcohol, Methylparaben, Butylparaben, Ethylparaben, Isobutylparaben, Propylparaben, Parfum (Fragrance), Linalool, Citronellol, Alpha-Isomethyl Ionone, Butylphenyl Methylpropional, Limonene, Benzyl Salicylate	For face, body and hands. With jojoba oil and vitamin E Dermatalogically approved	Perfumed, white – opaque, less runny than E45	200mL tub, white, opaque package	£2.98 (special offer save £1)
Basics Hand and Body Lotion	The Boots Company PLC, Nottingham, England, NG2 3AA	Aqua, Paraffinum liquidum, Hydrogenated vegetable glycerides citrate, Phenoxyethanol, Carbomer, Methylparaben, Tetrasodium EDTA, Potassium hydroxide, Butylparaben, Ethylparaben, Isobutylparaben, Propylparaben	Moisturises and softens your skin	Unperfumed, white - opaque, quite runny	500mL large, translucent bottle	61p

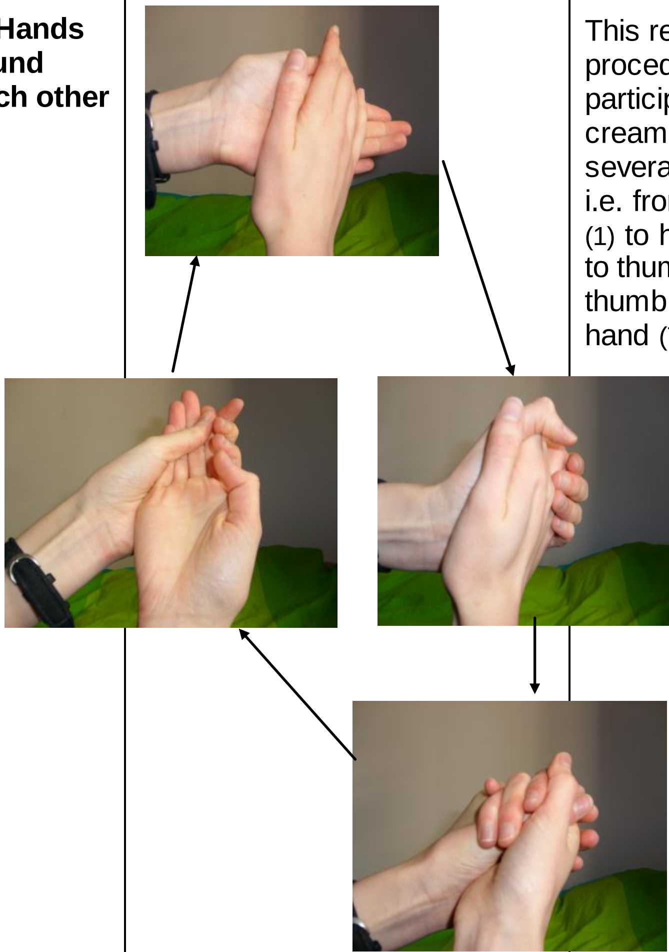
Mango Body Butter	The Body Shop Int. Plc, BN17 6LS, UK	Water, Mangifera Indica (Mango) Seed Oil, Prunus Amygdalus Dulcis (Sweet Almond) Oil, Theobroma Cacao (Cocoa) Butter, Glycerin, Cyclomethicone, Glyceryl Stearate, PEG-100 Stearate, Cetearyl Alcohol, Lanolin Alcohol, Phenoxyethanol, Fragrance, Methylparaben, Propylparaben, Xanthan Gum, Benzyl Alcohol, Disodium EDTA, Hexyl Cinnamal, Linalool, Amyl Cinnamal, Benzyl Benzoate, Limonene, Potassium Hydroxide, Sodium Hydroxide, Citral, Eugenol, Farnesol, Geraniol, Citronellol, Annatto.	With mango seed oil to moisturise. VERY DRY SKIN	Perfumed, yellow, medium thickness	200mL, bright orange, opaque tub	£12
Neutrogena concentrated hand cream	Neutrogena Div – Johnson & Johnson Consumer France s.a.s, 92787 Issy Cdx 9 (Paris) France.	Aqua, Glycerin, Cetearyl Alcohol, Stearic Acid, Palmitic Acid, Sodium Cetearyl Sulfate, Dilauryl Thiodipropionate, Sodium Sullfate, Methylparaben, Propylparaben, Parfum – [FPT0381]	Norwegian Formula. Instantly relieves dry or chapped hands. Just a dab needed. Dermatolo-gist tested.	Perfumed. Translucent, gel like appearance, like Vaseline petroleum jelly – reasonably viscous.	50mL tube, white matt finish, opaque	£3.38

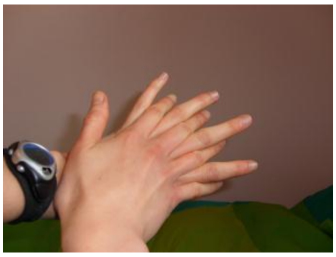
Dove regenerating hand cream	Unilever UK, Freepost, Admail 1000, London SW1A 2XX	Aqua, Glycerin, Cetyl Alcohol, Octyldodecanol, Stearic Acid, Hydrogenated Polydecene, Dimethicone, Butyrospermum Parkii, Acacia Senegal, Synthetic Wax, Isohexadecane, Propylene Glycol, Sorbitan Laurate, Glycol Stearate, Glyceryl Stearate, Polysorbate 80, Stearamide AMP, Dicaprylyl Carbonate, Calcium Chloride, Acrylates/C10-30 Alkyl Acrylate Cross-polymer, Xanthan Gum, Sodium Acrylate/Acryloyldimethyl Taurate Copolymer, Gelatin, Parfum, Triethanolamine, Sorbitan Oleate, Diazolidinyl Urea, Phenoxyethanol, Methylparaben, Propylparaben, Alpha-Isomethyl Ionone, Butylphenyl Methylpropional, Citronellol, Geraniol, Hexyl Cinnamal, Hydroxycitronellal, Hydroxyisohexyl 3-Cyclohexene Carboxaldehyde, Limonene, Linalool, CI 77007, CI 77891.	Night Care with Vitamin F. Strengthens the skin barrier, improves skin firmness & resilience, hands regain their natural beauty, wake up to softer smoother hands, instantly absorbed. Dermatologically tested.	Perfumed, white opaque cream, medium viscosity. Blue bits in it	75mL tube – blue matt finish, opaque	£2.29
Vaseline Intensive Care healthy hand and nail everyday care lotion	Unilever UK, Freepost, Admail 1000, London SW1A 2XX	Aqua, Paraffinum Liquidum, Stearic Acid, Dimethicone, Glycerin, Glycol Stearate, Sodium PCA, Lactic Acid, Potassium Lactate, Urea, Collagen Amino Acids, Tocopheryl Acetate, Retinyl Palmitate, Helianthus Annus Seed Oil, Sodium Stearoyl Lactylate, Lecithin, Hydrolyzed Keratin, Hydrolyzed Milk Protein, Cyclopentasiloxane, Triethanolamine, Glyceryl Stearate, Stearamide AMP, Cetyl Alcohol, Magnesium Aluminium Silicate, Carbomer, Disodium EDTA, Parfum, Phenoxyethanol, Methylparaben, Propylparaben, Benzyl Benzoate, Benzyl Salicylate, Butylphenyl Methylpropional, Citronellol, Geraniol, Hexyl Cinnamal, Hydroxycitronellal, Hydroxyisohexyl 3-Cyclohexene Carboxaldehyde, Limonene, Linalool, CI 16255.	Softens hands and strengthens nails by 50%. With Keratin, milk proteins & vitamin E	Perfumed, quite runny, pinky colour	200mL, opaque light pink and dark blue bottle	£2.99

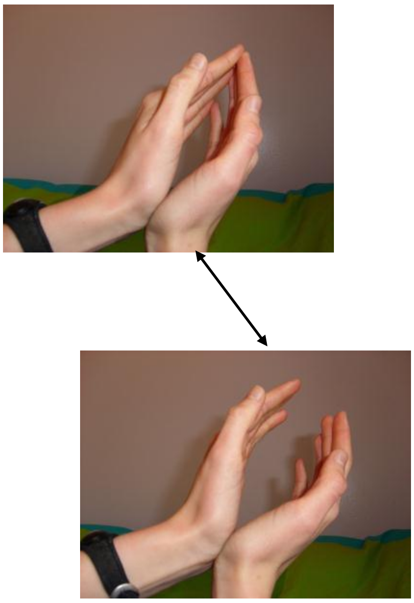
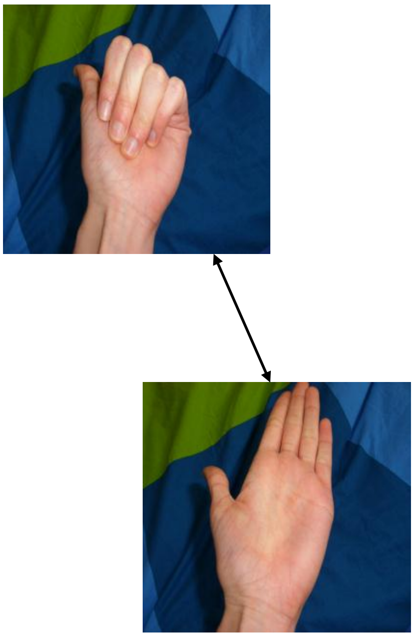
Appendix II: Skin cream application procedures observed during focus groups

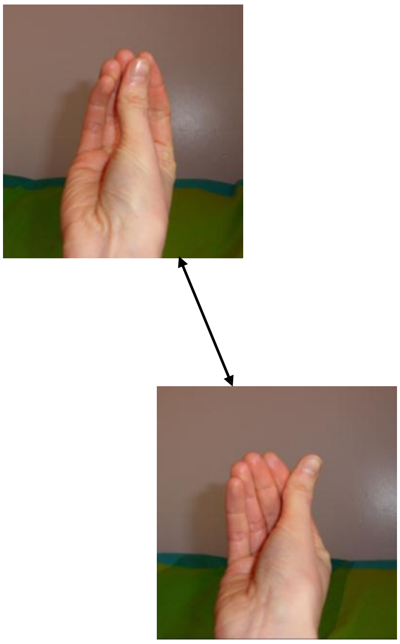
***Table A2.1**: Common skin cream application methods observed during the focus groups. In the right hand column a star rating for each named procedure has been given where six stars (******) represents the most common methods and one star (*) the least common.*

NAME	PICTURE	DESCRIPTION	
1) Palm to palm		Rubbing hands in circular motion palm to palm or straight up and down.	******
2) Palm to back		Rubbing palm over back of hand with either wiping or rubbing motion.	****
3) Holding hands *(Clasped hands like holding)*		This refers to the case when subjects hold their own hands (like they are holding someone else's) either in sequence as part of the rubbing in procedure e.g. from 'palm to palm' (1) to holding hands or they hold hands for a short while.	**
4) Palms to knuckles and finger tips		Rubbing hands together and holding like the picture for a few seconds.	*

5) Hands round each other		This refers to the procedure when participants apply the cream by following several hand positions i.e. from palm to palm (1) to holding hands (3) to thumb clasps (6) to thumb use on back of hand (7).	******
6) Thumb clasp		Clasping the thumb as part of hands round each other rubbing in motion (5). Hands never stay clasped to thumb for a long time this is more of a smooth motion e.g. go from palm to palm (1) to hold hands (3) to thumb clasp (6).	***
7) Use of thumb on back of hand		As part of the rubbing in method the thumb is used to massage/rub the cream into the back of the hand.	***

8) Use of thumb on palm		As part of the rubbing in method, the thumb is used to massage cream into palm.	***
9) Wrist		This picture refers to the fact that the subject rubs the cream in right down to and including their wrists.	***
10) Finger webs	 	As part of rubbing in cream regime include finger webs either as distinct rubbing or as part of hands round each other motion (5).	****
11) Use of finger to rub in		This refers to the subject rubbing in the cream using one finger for a short while. This is usually observed at the beginning following initial application of cream to finger tip, back of hand or palm.	******

12) Tap hands		Tapping of hands following application of cream.	**
13) Clasp to fist and back		This refers to when the panellist clasps their fingers to palm and releases. It is thought that this is some sort of tackiness test.	*****

14) Rubbing in bread method		Use thumb to move over fingers like rubbing in bread method.	***
15) Around cuticles		Rub in cream round cuticles using fingers or thumb to massage in.	***

Appendix III: Results from rating 'initial greasiness' vs. 'slipperiness'

Descriptive profiling of skin creams results. Graphs show results from rating of 3 skin creams for the attributes 'initial greasiness' and 'slipperiness'. Samples are given on the x-axis; the y-axis represents the intensity at which samples were rated for that attribute; the different coloured lines represent the different panelists - panellist's names have been removed from the graph for confidentiality. Only results for 6 panellists are shown since some panellists were unable to attend every training session.

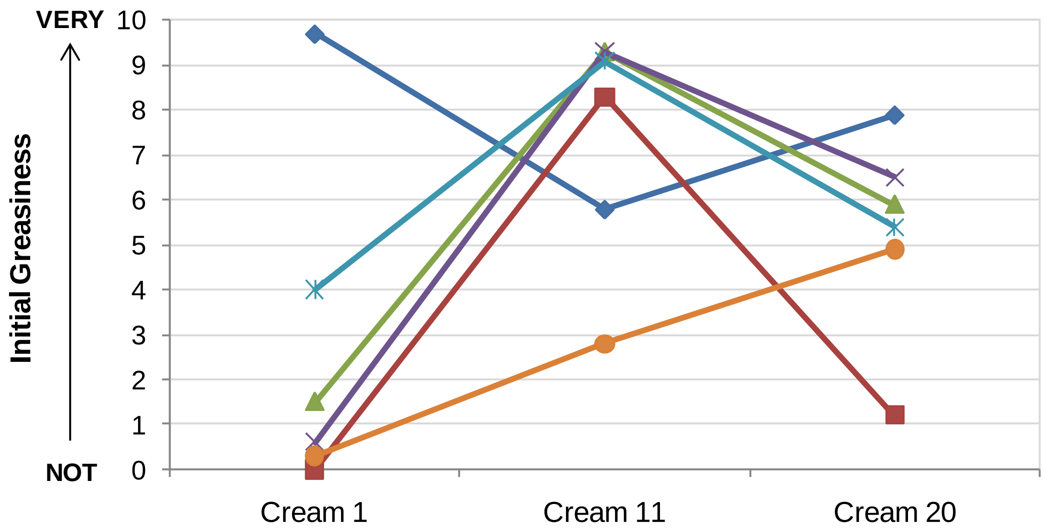


Figure A3.1: *Rating the attribute 'Initial Greasiness'.*

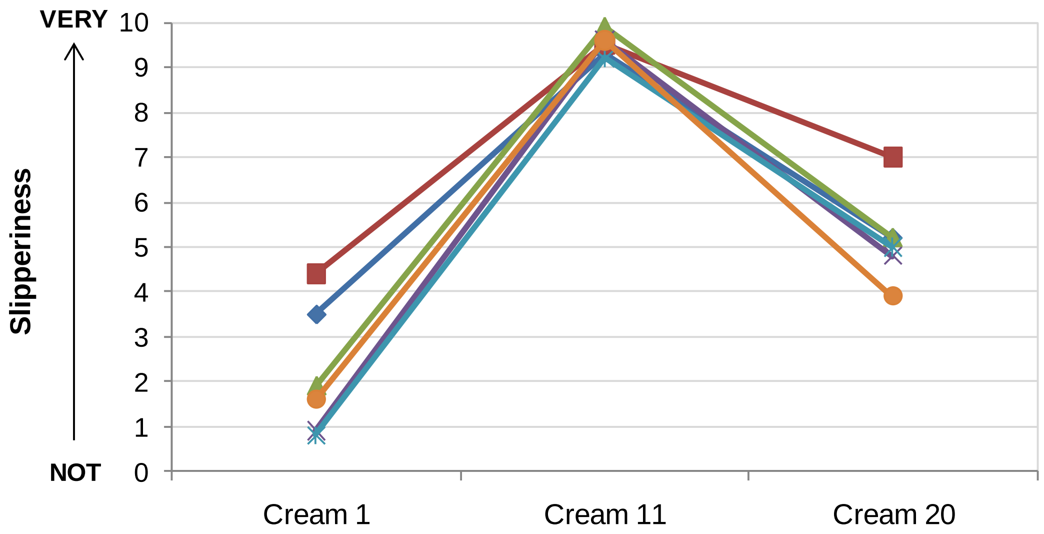


Figure A3.2: *Rating the attribute 'Slipperiness'.*

Appendix IV: Discrimination ability of panellists as determined during preliminary rating of 8 samples in triplicate

Table A4.1: *ANOVA results for individual panellists as obtained during preliminary rating of a subset of 8 model skin cream samples in triplicate for the sensory attributes. Non significant differences (p > 0.05) are given in bold type indicating lack of discrimination ability.*

Judge	FIRMNESS	THICKNESS	RESISTANCE	SPREADABILITY	STICKINESS	COOLING	DRYING	DRAGGING	SLIPPERINESS	ABSORPTIOIN	FINAL GREASINESS
A	0.000	0.000	0.000	0.000	0.000	0.001	0.001	0.000	0.000	0.000	0.003
B	0.000	0.000	0.000	0.000	**0.917**	**0.208**	0.000	0.000	0.000	0.000	0.003
C	0.000	0.000	0.000	0.000	0.018	0.000	0.000	0.001	0.001	0.001	0.000
D	0.000	0.000	0.000	0.000	0.006	**0.625**	**0.144**	**0.191**	0.000	**0.191**	0.000
E	0.000	0.000	0.000	0.000	0.004	0.026	0.000	0.000	0.000	0.000	0.005
F	0.000	0.000	0.000	0.000	0.048	0.000	0.003	0.021	0.000	0.021	0.000
G	0.000	0.000	0.000	0.000	0.004	**0.116**	0.028	0.003	0.005	0.003	0.024
H	0.000	0.000	0.000	0.000	0.000	**0.056**	0.000	0.001	0.000	0.001	0.000
I	0.000	0.000	0.000	0.001	0.011	**0.113**	0.011	0.038	0.002	0.038	0.000
J	**0.270**	**0.272**	**0.698**	**0.064**	**0.449**	**0.925**	**0.122**	**0.336**	**0.045**	**0.336**	**0.614**

Table A4.2: *Tukeys HSD results showing the number of homogeneous subsets individual panellists separated samples into for the different attributes during preliminary rating of a subset of 8 model skin cream samples in triplicate. Note that 8 subsets would be the maximum number possible (good discrimination ability) and 1 the minimum number (indicating lack of discrimination ability).*

Judge	FIRMNESS	THICKNESS	RESISTANCE	SPREADABILITY	STICKINESS	COOLING	DRYING	DRAGGING	SLIPPERINESS	ABSORPTIOIN	FINAL GREASINESS
A	3	4	4	4	4	3	3	3	3	3	2
B	2	2	4	3	**1**	**1**	4	3	3	2	2
C	5	3	3	3	2	2	3	3	2	3	2
D	3	3	3	4	2	**1**	**1**	**1**	3	2	2
E	5	4	4	3	2	**1**	3	3	3	3	2
F	4	5	3	3	**1**	3	3	2	3	2	3
G	4	3	3	3	3	**1**	**1**	3	2	2	2
H	3	4	4	3	3	**1**	3	3	3	5	3
I	4	3	3	3	2	**1**	2	**1**	3	3	2
J	?	?	?	?	?	?	?	?	?	?	?

Appendix V: Comparing mean results obtained during rating absorption for 40 creams 3 replicates and the 12 consumer study creams 3 replicates

Note that Figure A5.1 includes 10 panellists and Figure A5.2 includes 8 panellists since only 8 of the trained panel were available to participate in the rating of the consumer study creams.

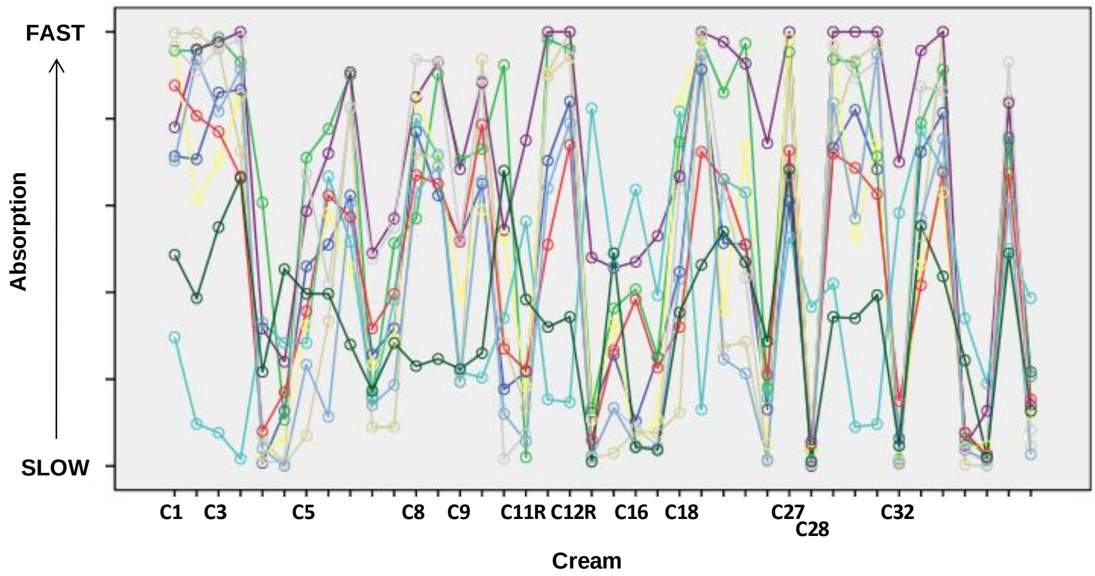


Figure A5.1: *Mean panelist scores as obtained for rating the attribute absorption for 40 creams with 3 replicates (consumer study creams are labelled for comparison).*

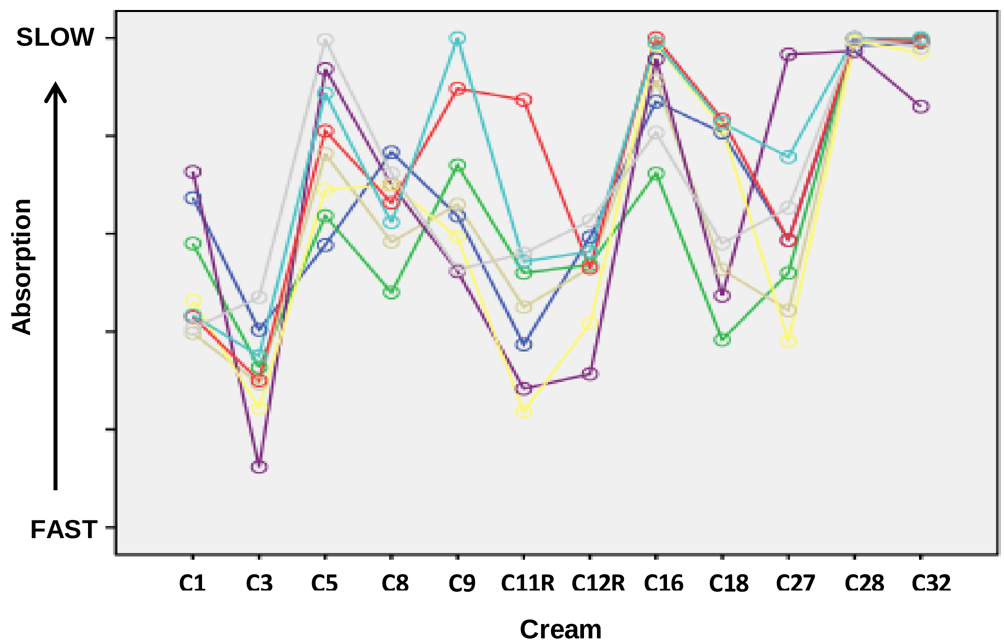


Figure A5.2: *Mean panelist scores as obtained for rating the attribute absorption for 12 creams with 3 replicates.*

Appendix VI: Pre-screening questionnaire used in consumer study recruitment

CONSUMER STUDY QUESTIONNAIRE

Background Information

This questionnaire is designed to provide us with more information about general consumers that use hand cream (to accompany the sensory test). Please fill in the questionnaire indicating which answers apply to you (underline or circle your response). Answers will be treated as confidential and any publication of overall results will have names removed.

Name:

Age:	15-20	21-25	26-30	31-35
	36-40	41-55	56-60	60+

Gender: Male Female

1) Are you allergic to any common ingredients found in hand creams?
(if yes please specify the ingredient)

Yes Ingredient(s):

No

2) Do you use hand creams (i.e. moisturisers/body lotion)?

Yes No

3) What make of skin cream do you generally use?
(please give name and make if possible)

4) How often do you use skin creams?

More than 3 times a day
2-3 times a day
Once a day
Once a week
Less than once a week

5) When purchasing skin cream, which skin type do you generally buy for?

Dry Oily Normal Other…..

Thank you for your participation, your help is appreciated!

Appendix VII: Gap correction explanation for narrow gap measurements

When carrying out narrow gap measurements, data needs correcting because results assume parallel plates are 100 % smooth and 100 % parallel at the set gap height (e.g. 50 μm gap) whereas in reality this is unlikely to be true (the gap may in fact be 70-80 μm). If data is not corrected, the viscosity would appear lower than it should – the fact that results are affected highlights the importance of gap correction, first considered by Kramer et al. (1987). Therefore to include this correction in our data we start by defining the corrected gap height, h_r , as

$$h_r = h_s + \varepsilon \qquad \text{(A7.1)}$$

where h_s is the gap height set within the computer software and ε is the gap error.

We know that the corrected shear rate, $\dot{\gamma}_r$ is given by

$$\dot{\gamma}_r = \frac{R\omega}{h_r} \qquad \text{(A7.2)}$$

where *R* is the radius of the parallel plates, ω is the angular velocity. We find by substituting Equation (A7.1) into Equation (A7.2) that

$$\dot{\gamma}_r = \frac{R\omega}{h_s + \varepsilon} \qquad \text{(A7.3)}$$

The shear stress, τ , is given by

$$\tau = \eta \dot{\gamma}_r \qquad \text{(A7.4)}$$

where η is the viscosity of the sample. Therefore by substitution of Equation (A7.3) into Equation (A7.4) we find

$$\tau = \eta \frac{R\omega}{h_s + \varepsilon} \qquad \text{(A7.5)}$$

With reference to Equation (A7.2) we know that

$$R\omega = \dot{\gamma}_s h_s \qquad \text{(A7.6)}$$

where $\dot{\gamma}_s$ is the shear rate set within computer, therefore we obtain

$$\tau = \eta \frac{\dot{\gamma}_s h_s}{h_s + \varepsilon} \qquad \text{(A7.7)}$$

Equation (A7.7) can be rearranged to find the following expression used to calculate the gap error

$$\frac{h_s \dot{\gamma}_s}{\tau} = \frac{1}{\eta} h_s + \frac{\varepsilon}{\eta} \qquad \text{(A7.8)}$$

To find the gap error we used a sample of silicone oil (a Newtonian fluid) and found the shear stress for increasing shear rate and repeated the experiment for gap heights of 50, 125, 250, 500 and 1000 μm. The gap error is then found by plotting $\frac{h_s \dot{\gamma}_s}{\tau}$ against h_s , then using linear regression to find the slope of the line and the y axis intercept as in the example in Figure A7.1. Kramer regression must be carried out each time the geometry of the machine is changed when carrying out narrow gap measurements.

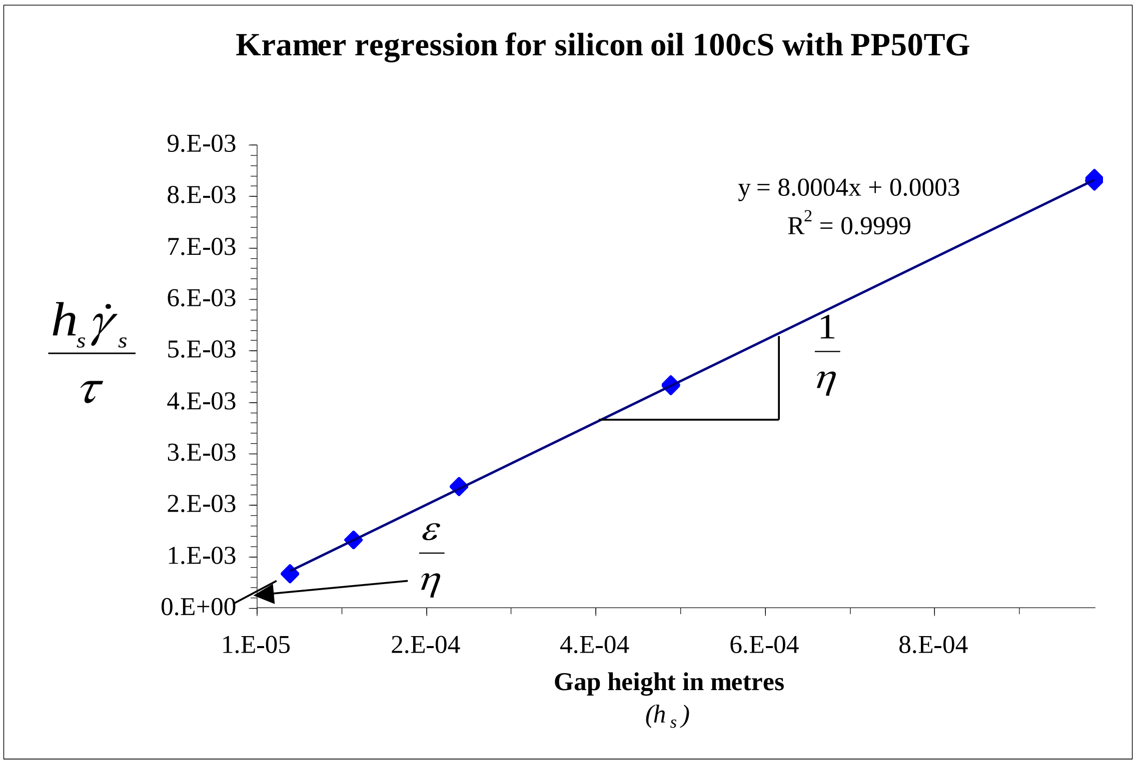


Figure A7.1: *Example of Kramer regression*

Once the gap error is known we use can use it to find the corrected shear rate and correct viscosity for the sample. From substitution of Equation (A7.6) into Equation (A7.3) and then rearranging we find that the corrected shear rate is given by

$$\dot{\gamma}_r = \frac{\dot{\gamma}_s}{1 + \varepsilon / h_s} \qquad \text{(A7.9)}$$

We can also rearrange Equation (A7.8) to find the following expression for the corrected viscosity.

$$\eta = \frac{\tau}{\dot{\gamma}_s}\left(1+\frac{\varepsilon}{h_s}\right) \qquad \text{(A7.10)}$$

Skin creams however are non-Newtonian so a further correction of 4/5 to the shear rate is required. This is implemented in the Equation for the viscosity (A7.11)

$$\eta = \frac{\tau}{4/5\,\dot{\gamma}_s}\left(1+\frac{\varepsilon}{h_s}\right) \qquad \text{(A7.11)}$$

The 4/5ths correction is for <u>non-Newtonian</u> fluids only. The bracketed section allows for gap errors.

The 4/5ths correction was derived by Shaw (2006), previously in 1987 Cross and Kaye reported a correction of 3/4ths, this correction was developed because the parallel plate geometry, although highly flexible, allows non-uniform flow to occur which is undesirable when measuring non-Newtonian substances that show non-linear response. Cross and Kaye's solution assumes the sample is Newtonian but the resulting shear rate assigned to the observed "Newtonian" viscosity is 3/4ths the rim shear rate. Gaussion integration over radius of the nonlinear stress profile creates this shift factor (Cross and Kaye, 1987).

Shaw & Liu (2006) re-assessed this theory and found that a shift factor of 0.8 (4/5ths) appears more accurate than that previously recorded 0.75 (3/4ths), showing < 1 % error rather than 3 % error. This 4/5ths correction is based on the 3rd moment of Gaussion approximation of the integral expression for torque.

Appendix VIII: Oscillation amplitude sweep summary table

Table A8.1: *Oscillation amplitude sweep summary table including yield stress and yield strain data.*

CREAM	Yield Stress		Strain for YS	
	Average	CV	Average	CV
	[Pa]	[%]	[-]	[%]
C1	297	5.5	0.9	0.0
C2	5	7.9	0.9	0.0
C3	48	9.2	0.7	0.0
C3R	57	2.7	1.1	0.0
C4	158	4.3	0.7	0.0
C4R	174	4.5	0.7	0.0
C5	2174	1.6	1.1	0.0
C5R	1438	0.7	1.1	0.0
C6	75	5.6	0.3	0.0
C7	658	10.9	0.3	0.0
C7R	592	11.0	0.2	0.1
C8	28	42.9	1.1	0.0
C8R	27	10.5	1.1	0.1
C9	328	6.2	0.7	0.0
C10	28	9.7	0.4	0.0
C11	15	4.8	1.5	0.0
C11R	6	11.7	1.1	0.0
C12	1326	4.4	0.2	0.0
C12R	1554	1.5	0.3	0.0
C14	1090	9.9	0.9	0.0
C15	1115	3.1	1.1	0.0
C16	505	7.6	0.9	0.0
C17	774	5.4	0.5	0.0
C18	669	3.3	0.3	0.0
C20	494	2.8	0.3	0.0
C23	699	2.9	0.7	0.0
C24	904	3.5	0.7	0.0
C25	49	3.1	1.1	0.1
C27	1796	5.1	1.1	0.0
C28	185	1.1	0.5	0.0
C29	307	19.8	0.5	0.0
C30	1276	16.2	0.7	0.0
C31	10	4.6	1.1	0.0
C32	362	14.0	0.7	0.0
C33	589	1.7	1.1	0.0
C34	365	11.5	0.5	0.0
C35	241	6.8	0.7	0.0
C36	99	2.1	0.5	0.0
C37	99	1.6	0.5	0.1
C40	1300	7.6	0.5	0.0

Table A8.2: *Oscillation amplitude sweep summary table including average and CV values for G', G'' and η* at 0.1 % strain for 40 model skin creams.*

CREAM	G′ at 0.1% strain		G′′ at 0.1% strain		η* at 0.1% strain	
	Average	CV	Average	CV	Average	CV
	[Pa]	[%]	[Pa]	[%]	[Pa.s]	[%]
C1	6422	15.7	2869	15.9	7034	15.7
C2	41	23.0	8	39.7	42	23.2
C3	528	8.9	142	14.4	547	9.2
C3R	661	7.2	194	15.3	689	7.8
C4	990	21.6	277	28.7	1028	22.1
C4R	1156	12.3	375	12.3	1215	12.1
C5	12257	7.3	4207	6.3	12960	7.2
C5R	8116	2.2	2805	4.8	8588	2.4
C6	797	4.5	160	3.9	813	4.4
C7	6495	5.0	2276	11.4	6883	5.6
C7R	7511	10.8	3069	16.4	8115	11.5
C8	236	57.5	55	70.8	243	58.2
C8R	234	18.8	44	20.7	238	18.7
C9	5293	20.0	1817	24.1	5597	20.4
C10	326	5.5	87	10.4	337	5.7
C11	100	18.4	26	24.5	103	18.5
C11R	40	20.2	8	55.8	40	20.0
C12	17887	2.2	5316	3.5	18660	2.3
C12R	17380	3.4	4657	5.4	17995	3.6
C14	57480	13.1	38039	13.7	68931	13.2
C15	5827	6.5	1327	8.1	5977	6.4
C16	2067	12.2	504	7.4	2128	12.0
C17	7759	6.7	3574	7.3	8543	6.7
C18	6409	8.6	1833	9.4	6666	8.7
C20	6415	7.4	2140	5.9	6763	7.2
C23	12024	8.6	4370	6.9	12797	8.4
C24	6185	5.3	1108	8.5	6284	5.4
C25	323	10.3	146	11.4	354	10.2
C27	9101	10.8	1963	13.1	9311	10.9
C28	1832	4.9	719	6.8	1968	5.1
C29	5891	30.6	1778	34.1	6154	30.9
C30	25848	65.8	8453	83.2	27291	66.8
C31	68	20.0	19	28.7	70	20.5
C32	7836	28.9	3370	29.3	8524	28.9
C33	1867	5.0	127	5.0	1871	4.9
C34	8779	31.8	3375	37.2	9410	32.5
C35	1922	20.1	561	39.3	2006	21.2
C36	1259	8.1	552	7.2	1375	7.8
C37	795	1.6	197	8.1	819	2.0
C40	10535	7.9	4061	7.3	11288	7.8

Table A8.3: *Oscillation amplitude sweep summary table including average and CV values for G', G'' and η* at 1 % strain for 40 model skin creams.*

CREAM	G′ at 1% strain		G″ at 1% strain		η* at 1% strain	
	Average	CV	Average	CV	Average	CV
	[Pa]	[%]	[Pa]	[%]	[Pa.s]	[%]
C1	4071	11.6	1776	11.6	4442	11.6
C2	42	23.0	9	27.8	43	23.2
C3	528	8.8	126	10.3	543	8.8
C3R	633	6.2	160	10.5	653	6.5
C4	1006	14.3	254	16.7	1037	14.4
C4R	1153	7.3	347	8.5	1202	7.4
C5	11818	3.7	3667	2.4	12375	3.6
C5R	7630	0.7	2409	1.2	8001	0.7
C6	738	6.3	132	8.0	749	6.3
C7	6575	3.8	2081	6.6	6897	4.0
C7R	7447	9.2	2782	15.4	7952	10.0
C8	223	57.0	48	72.6	228	57.7
C8R	214	18.4	36	22.3	217	18.4
C9	4033	18.2	1229	22.8	4216	18.6
C10	309	8.7	73	9.7	318	8.7
C11	98	19.0	26	20.2	101	19.0
C11R	40	20.6	9	25.8	41	20.9
C12	17586	3.5	4607	3.8	18179	3.5
C12R	16968	1.4	4066	1.9	17450	1.4
C14	19438	13.6	11122	12.5	22399	13.2
C15	5474	7.0	1091	2.6	5581	6.8
C16	2011	6.5	425	4.2	2056	6.3
C17	8021	3.8	3487	3.7	8745	3.7
C18	6673	5.6	1710	7.4	6889	5.7
C20	6756	6.9	1994	9.8	7044	7.1
C23	11006	1.7	3827	4.3	11653	2.0
C24	5935	4.7	919	6.9	6006	4.7
C25	310	3.1	128	7.4	335	3.6
C27	8541	7.5	1581	8.4	8686	7.5
C28	1712	3.0	623	4.1	1822	3.1
C29	4280	27.8	1250	35.1	4460	28.4
C30	13112	45.8	3543	68.4	13614	47.0
C31	68	20.1	18	23.4	70	20.3
C32	5465	21.5	2121	22.6	5862	21.6
C33	1847	2.1	132	3.7	1851	2.1
C34	5494	24.5	2085	31.8	5878	25.3
C35	1766	14.1	411	13.9	1814	14.1
C36	1161	9.3	473	9.2	1254	9.2
C37	814	5.4	190	12.3	836	5.8
C40	10229	3.2	3647	4.0	10858	3.2

Table A8.4: *Oscillation amplitude sweep summary table including average and CV values for G' G" and η* at 100 % strain for 40 model skin creams.*

CREAM	G′ at 100% strain		G″ at 100% Strain		η* at 100% strain	
	Average	CV	Average	CV	Average	CV
	[Pa]	[%]	[Pa]	[%]	[Pa.s]	[%]
C1	301	5.5	289	6.2	418	5.7
C2	5	7.5	5	13.6	7	9.9
C3	48	9.0	43	9.2	65	9.1
C3R	58	2.6	51	3.3	77	2.7
C4	158	3.4	149	6.7	217	4.9
C4R	176	4.4	174	3.6	248	4.0
C5	2213	1.8	1772	2.9	2836	2.2
C5R	1464	0.7	1130	1.2	1850	0.8
C6	68	6.1	86	5.7	110	5.7
C7	484	11.5	673	8.4	830	9.5
C7R	463	8.4	663	6.2	810	6.8
C8	28	44.3	27	52.2	39	48.0
C8R	27	11.0	24	16.6	36	13.3
C9	330	5.4	360	14.3	489	10.1
C10	27	9.0	29	8.2	39	8.6
C11	15	6.3	12	13.3	19	8.9
C11R	6	12.0	6	16.8	9	13.8
C12	1091	2.6	1577	2.7	1919	2.1
C12R	1245	2.3	1660	0.9	2076	1.4
C14	1111	9.5	949	12.4	1462	10.5
C15	1131	3.2	827	4.9	1401	3.7
C16	510	7.5	368	7.0	629	7.3
C17	717	7.1	980	4.9	1215	5.1
C18	549	1.6	784	3.9	958	3.2
C20	441	2.8	608	4.7	751	4.0
C23	698	2.7	860	2.2	1107	2.3
C24	910	3.6	742	4.9	1175	4.1
C25	50	3.1	44	5.5	67	4.1
C27	1829	5.1	1115	7.2	2142	5.7
C28	163	0.2	211	2.6	267	1.7
C29	291	17.6	334	22.5	444	20.2
C30	1263	15.7	854	17.2	1527	15.7
C31	10	4.7	8	12.1	13	7.1
C32	367	13.3	452	18.2	583	15.8
C33	595	1.7	289	2.9	662	1.9
C34	336	4.7	360	15.7	494	9.9
C35	236	5.7	224	9.9	326	6.3
C36	96	0.7	119	3.4	153	2.0
C37	97	2.3	82	0.4	128	1.3
C40	1253	8.3	1392	5.1	1874	6.5

Table A8.5: *Oscillation amplitude sweep summary table including average and CV values for tanδ at 0.1 %, 1 % and 100 % strain for 40 model skin creams.*

CREAM	tanδ at 0.1% strain		tanδ at 1% Strain		tanδ at 100% strain	
	Average	CV	Average	CV	Average	CV
	[Pa]	[%]	[Pa]	[%]	[Pa.s]	[%]
C1	0.45	22.4	0.44	16.4	0.96	8.3
C2	0.21	45.9	0.20	36.1	0.90	15.6
C3	0.27	16.9	0.24	13.5	0.89	12.9
C3R	0.29	16.9	0.25	12.3	0.87	4.2
C4	0.28	35.9	0.25	22.0	0.94	7.5
C4R	0.32	17.4	0.30	11.2	0.99	5.7
C5	0.34	9.7	0.31	4.5	0.80	3.4
C5R	0.35	5.3	0.32	1.4	0.77	1.4
C6	0.20	6.0	0.18	10.2	1.26	8.4
C7	0.35	12.4	0.32	7.6	1.39	14.3
C7R	0.41	19.6	0.37	18.0	1.43	10.4
C8	0.23	91.2	0.22	92.3	0.95	68.5
C8R	0.19	27.9	0.17	28.9	0.90	19.9
C9	0.34	31.3	0.30	29.2	1.09	15.2
C10	0.27	11.7	0.23	13.0	1.08	12.2
C11	0.27	30.7	0.27	27.7	0.79	14.7
C11R	0.19	59.3	0.24	33.0	0.90	20.7
C12	0.30	4.1	0.26	5.2	1.45	3.7
C12R	0.27	6.4	0.24	2.4	1.33	2.5
C14	0.66	18.9	0.57	18.5	0.85	15.6
C15	0.23	10.4	0.20	7.4	0.73	5.8
C16	0.24	14.3	0.21	7.7	0.72	10.2
C17	0.46	9.9	0.43	5.3	1.37	8.6
C18	0.29	12.8	0.26	9.3	1.43	4.2
C20	0.33	9.5	0.30	12.0	1.38	5.4
C23	0.36	11.0	0.35	4.6	1.23	3.5
C24	0.18	10.0	0.15	8.3	0.82	6.1
C25	0.45	15.3	0.41	8.0	0.89	6.3
C27	0.22	17.0	0.19	11.3	0.61	8.9
C28	0.39	8.4	0.36	5.1	1.29	2.6
C29	0.30	45.8	0.29	44.8	1.15	28.5
C30	0.33	106.1	0.27	82.3	0.68	23.3
C31	0.27	35.0	0.26	30.9	0.86	13.0
C32	0.43	41.2	0.39	31.2	1.23	22.6
C33	0.07	7.1	0.07	4.3	0.49	3.4
C34	0.38	49.0	0.38	40.1	1.07	16.4
C35	0.29	44.2	0.23	19.8	0.95	11.4
C36	0.44	10.8	0.41	13.1	1.24	3.5
C37	0.25	8.3	0.23	13.4	0.85	2.3
C40	0.39	10.8	0.36	5.1	1.11	9.7

Appendix IX: Oscillation frequency sweep summary table

Table A9.1: *Oscillation frequency sweep summary table including the magnitude of the slopes logG'-logω and logG"-logω, the intercepts and R^2 values.*

CREAM	logG′-logω			logG″-logω		
	Slope	Intercept	R^2	Slope	Intercept	R^2
C1	0.15	3.66	0.99	0.09	3.30	0.98
C2	0.15	1.65	1.00	0.17	1.01	0.93
C3	0.17	2.70	1.00	0.08	2.12	0.92
C3R	0.17	2.81	0.99	0.06	2.23	0.92
C4	0.18	3.07	0.99	0.15	2.50	0.98
C4R	0.20	3.05	1.00	0.18	2.54	0.98
C5	0.19	4.02	1.00	0.13	3.54	0.98
C5R	0.18	3.93	1.00	0.12	3.45	0.97
C6	0.11	2.79	0.99	-0.01	2.07	0.09
C7	0.19	3.76	1.00	0.14	3.25	0.99
C7R	0.24	3.89	1.00	0.17	3.47	1.00
C8	0.13	2.37	1.00	0.05	1.72	0.76
C8R	0.10	2.20	0.99	0.03	1.45	0.23
C9	0.13	3.61	1.00	0.08	3.06	0.88
C10	0.14	2.47	1.00	0.12	1.84	0.84
C11	0.16	2.04	1.00	0.15	1.48	0.92
C11R	0.16	1.66	1.00	0.21	1.07	0.93
C12	0.18	4.24	0.99	0.04	3.68	0.96
C12R	0.17	4.23	0.99	0.03	3.65	0.83
C14	0.08	4.29	0.98	0.04	4.05	0.50
C15	0.14	3.66	0.99	0.05	3.00	0.78
C16	0.13	3.32	1.00	0.04	2.67	0.67
C17	0.27	3.93	1.00	0.25	3.59	1.00
C18	0.17	3.83	1.00	0.09	3.26	0.97
C20	0.19	3.81	1.00	0.09	3.29	0.98
C23	0.17	3.99	1.00	0.12	3.52	0.96
C24	0.10	3.74	1.00	0.02	2.98	0.22
C25	0.23	2.48	1.00	0.23	2.08	0.98
C27	0.12	3.93	0.99	-0.03	3.23	0.46
C28	0.23	3.23	1.00	0.17	2.81	0.99
C29	0.12	3.49	0.98	0.01	2.94	0.24
C30	0.02	4.08	0.52	-0.03	3.56	0.21
C31	0.16	1.83	1.00	0.15	1.24	0.92
C32	0.18	3.76	1.00	0.13	3.36	0.97
C33	0.04	3.28	0.99	-0.04	2.17	0.30
C34	0.13	3.71	0.99	0.08	3.26	0.93
C35	0.13	3.19	1.00	0.06	2.55	0.71
C36	0.22	3.00	1.00	0.17	2.58	0.99
C37	0.15	2.92	0.99	0.06	2.33	0.75
C40	0.22	4.03	1.00	0.22	3.59	1.00

Table A9.2: *Oscillation frequency sweep summary table including the average tanδ, G' and G'' data at 1 rad.s^{-1}.*

CREAM	tanδ at 1 rad.s^{-1}		G′ at 1 rad.s^{-1}		G″ at 1 rad.s^{-1}	
	Average	CV	Average	CV	Average	CV
	[-]	[%]	[Pa]	[%]	[Pa]	[%]
C1	0.42	20.3	4665	13.2	1961	15.4
C2	0.21	45.0	45	18.4	10	41.0
C3	0.25	25.7	506	17.8	128	18.6
C3R	0.25	14.3	657	7.8	167	11.9
C4	0.25	16.0	1211	11.9	305	10.8
C4R	0.30	17.5	1138	11.2	337	13.5
C5	0.32	20.4	10750	13.9	3413	14.9
C5R	0.33	18.3	8512	11.1	2797	14.6
C6	0.17	27.3	629	18.2	109	20.3
C7	0.30	9.2	5834	8.2	1764	4.2
C7R	0.38	4.7	7858	3.3	3014	3.3
C8	0.22	66.7	235	43.8	51	50.4
C8R	0.17	69.2	160	44.2	27	53.2
C9	0.28	2.8	4093	1.6	1128	2.3
C10	0.23	26.5	296	18.6	67	18.9
C11	0.26	10.8	110	9.4	28	5.4
C11R	0.24	15.2	45	6.5	11	13.8
C12	0.27	23.2	17775	16.1	4814	16.8
C12R	0.25	4.0	17310	2.7	4344	2.9
C14	0.54	20.1	19778	14.3	10612	14.2
C15	0.21	15.5	4649	12.6	981	9.0
C16	0.21	7.5	2125	6.9	451	2.9
C17	0.45	18.3	8589	11.7	3898	14.0
C18	0.26	3.8	6769	2.8	1771	2.6
C20	0.29	10.2	6644	4.5	1949	9.1
C23	0.33	9.8	9859	7.5	3225	6.3
C24	0.16	7.8	5532	5.7	898	5.4
C25	0.39	17.2	302	12.9	117	11.3
C27	0.19	10.1	8683	6.7	1611	7.5
C28	0.36	8.0	1751	5.1	628	6.2
C29	0.26	35.6	3191	21.2	839	28.7
C30	0.29	147.5	11923	72.1	3467	128.6
C31	0.24	10.3	68	7.7	16	6.9
C32	0.39	15.2	5791	10.3	2256	11.1
C33	0.07	4.4	1928	3.5	136	2.6
C34	0.34	26.2	5164	16.4	1765	20.4
C35	0.21	22.7	1563	10.2	335	20.3
C36	0.37	31.1	1030	16.7	380	26.2
C37	0.24	10.1	840	4.2	202	9.2
C40	0.37	9.0	10600	5.5	3875	7.1

Appendix X: Steady shear summary tables including Cross model and yield stress values

Table A10.1: *Cross model summary table containing the infinite and zero shear viscosities for the 40 model skin cream samples.*

CREAM	Infinite Shear Viscosity			Zero Shear Viscosity		
	Average	Median	CV	Average	Median	CV
	[Pa.s]	[Pa.s]	[%]	[Pa.s]	[Pa.s]	[%]
C1	0.110	0.129	41.4	98,116	102,110	21.7
C2	0.173	0.170	3.6	11,255	11,935	47.8
C3	0.210	0.208	9.9	15,529	15,563	6.7
C3R	0.162	0.162	0.2	17,603	17,603	4.4
C4	0.149	0.158	25.5	33,795	35,013	10.0
C4R	0.200	0.217	23.3	29,399	28,642	7.0
C5	0.000	0.000	70.9	266,263	267,890	4.6
C5R	0.001	0.000	199.9	214,743	183,215	32.7
C6	0.258	0.277	17.8	29,602	32,854	24.7
C7	0.251	0.280	26.8	163,450	155,750	8.3
C7R	0.117	0.102	96.9	166,235	168,390	5.0
C8	0.158	0.158	4.7	7,602	6,201	55.4
C8R	0.177	0.169	8.3	8,657	8,714	29.3
C9	0.072	0.076	26.3	133,990	132,205	7.0
C10	0.246	0.251	8.0	13,288	14,123	14.5
C11	0.236	0.237	2.0	4,784	4,755	3.9
C11R	0.209	0.209	2.8	7,578	8,335	44.8
C12	0.297	0.368	87.2	457,947	449,140	4.5
C12R	0.092	0.117	101.2	444,566	447,300	2.6
C14	0.000	0.000	25.7	627,838	640,475	37.7
C15	0.029	0.000	172.2	152,823	148,580	14.3
C16	0.354	0.341	51.8	65,472	65,245	8.0
C17	0.149	0.010	190.7	144,703	135,270	16.1
C18	0.119	0.121	46.6	197,103	193,690	5.8
C20	0.233	0.217	71.8	172,192	174,970	5.5
C23	0.000	0.000	37.7	293,897	300,760	5.8
C24	0.184	0.174	11.9	262,513	265,920	10.4
C25	0.308	0.328	12.3	6,898	7,143	6.4
C27	0.251	0.246	26.3	320,860	316,500	3.6
C28	0.130	0.129	1.2	32,906	33,879	13.2
C29	0.084	0.102	91.3	97,781	86,796	20.8
C30	0.514	0.481	52.9	489,172	487,920	10.2
C31	0.176	0.173	4.5	2,814	2,833	8.7
C32	0.003	0.000	200.0	135,075	138,305	8.1
C33	0.511	0.458	25.6	165,908	143,235	42.3
C34	0.150	0.147	15.0	159,678	165,395	14.5
C35	0.146	0.152	12.1	47,891	44,328	20.0
C36	0.155	0.152	6.8	17,694	19,742	50.3
C37	0.311	0.327	12.2	22,279	22,279	1.3
C40	0.000	0.000	18.0	225,130	222,150	3.9

Table A10.2: *Cross model summary table containing the Cross model a- and p-values for the 40 model skin cream samples.*

CREAM	CROSS model a value			CROSS model p value		
	Average	Median	CV	Average	Median	CV
	[s]	[s]	[%]	[-]	[-]	[%]
C1	203	212	13.8	0.924	0.923	1.0
C2	1065	1124	49.1	0.731	0.731	0.8
C3	170	161	11.2	0.857	0.855	1.5
C3R	192	192	2.6	0.903	0.903	0.2
C4	125	120	10.1	0.873	0.872	0.8
C4R	104	102	5.4	0.866	0.865	1.0
C5	86	86	1.4	0.927	0.929	0.5
C5R	97	85	25.0	0.923	0.922	0.9
C6	310	284	27.2	0.883	0.886	2.2
C7	164	173	11.2	0.942	0.935	1.6
C7R	142	146	10.6	0.955	0.956	0.6
C8	182	187	11.6	0.827	0.817	3.7
C8R	197	193	25.9	0.851	0.835	3.2
C9	258	257	4.9	0.897	0.898	0.6
C10	228	226	4.3	0.864	0.872	2.0
C11	147	150	6.4	0.790	0.792	1.0
C11R	597	683	43.5	0.713	0.714	0.5
C12	216	211	5.9	0.986	0.989	0.5
C12R	233	239	4.5	0.970	0.969	0.4
C14	512	516	35.1	0.918	0.923	1.6
C15	119	121	5.1	0.934	0.934	0.4
C16	91	92	1.4	0.944	0.943	1.0
C17	113	109	7.5	0.917	0.915	1.5
C18	201	202	2.8	0.944	0.945	0.3
C20	197	199	4.0	0.949	0.949	0.5
C23	298	295	8.0	0.916	0.920	1.1
C24	220	218	4.8	0.931	0.931	0.3
C25	69	74	15.2	0.836	0.839	1.0
C27	132	127	7.2	0.955	0.955	0.1
C28	112	110	4.0	0.926	0.926	0.2
C29	252	218	24.9	0.932	0.933	0.4
C30	324	320	13.7	0.948	0.948	1.7
C31	142	143	11.7	0.778	0.777	1.4
C32	226	225	5.5	0.903	0.903	0.7
C33	213	180	50.7	0.953	0.948	2.2
C34	322	327	10.3	0.942	0.943	0.6
C35	150	143	13.3	0.924	0.920	1.3
C36	143	144	4.5	0.921	0.921	0.7
C37	139	138	3.7	0.902	0.902	0.3
C40	121	115	8.7	0.925	0.927	0.4

Table A10.3: *Steady shear summary table containing the Cross model R-squared values and the yield stresses for all 40 samples. Note that the shear rate ranged between 0.0001 and 10,000s^{-1} for all samples.*

CREAM	CROSS MODEL R-SQUARED VALUES	YIELD STRESS		
	Average	Average	Median	CV
	[-]	[Pa]	[Pa]	[%]
C1	0.989	340	327	25.9
C2	0.990	4	4	8.8
C3	0.983	37	37	5.4
C3R	0.994	47	47	6.4
C4	0.996	123	124	8.9
C4R	0.996	122	118	9.0
C5	0.998	1480	1515	11.3
C5R	0.992	1062	1048	32.1
C6	0.992	45	41	24.4
C7	0.992	503	425	27.8
C7R	0.982	605	604	11.2
C8	0.998	21	16	61.9
C8R	0.996	20	19	20.0
C9	0.989	224	224	11.2
C10	0.990	24	26	20.8
C11	0.992	12	12	8.3
C11R	0.990	4	4	12.0
C12	0.989	1153	1160	2.7
C12R	0.991	1044	1033	4.8
C14	0.943	799	801	14.5
C15	0.997	511	520	3.7
C16	0.996	333	343	9.9
C17	0.980	553	598	26.8
C18	0.997	554	520	15.3
C20	0.990	435	446	8.7
C23	0.954	489	502	5.9
C24	0.997	606	614	8.4
C25	0.997	46	43	10.9
C27	0.997	1541	1528	13.8
C28	0.993	139	148	15.1
C29	0.969	168	167	15.5
C30	0.991	845	834	6.6
C31	0.985	7	7	5.4
C32	0.976	270	260	10.0
C33	0.982	433	434	6.5
C34	0.982	342	339	5.8
C35	0.993	140	141	5.7
C36	0.997	85	83	11.8
C37	0.988	73	73	1.4
C40	0.997	845	822	7.1

Appendix XI: Summary tables of rheology results for consumer study creams

Table A11.1: *Oscillation amplitude sweep summary table including yield stress and yield strain data for the consumer study skin creams.*

CREAM	Yield Stress		Strain for YS	
	Average	CV	Average	CV
	[Pa]	[%]	[-]	[%]
C1	108	16.6	1.15	0.1
C3	38	10.5	1.15	0.1
C5	1707	1.5	1.15	0.0
C8	57	37.6	0.87	0.0
C9	233	7.7	0.66	0.0
C11R	5	4.4	0.87	0.0
C16	601	4.8	0.87	0.0
C18	415	8.9	0.38	0.0
C12R	1060	1.9	0.16	0.0
C27	2001	7.8	0.66	0.0
C28	106	9.0	0.87	0.0
C32	405	13.5	0.50	0.0

Table A11.2: *Oscillation amplitude sweep summary table including average and CV values for G', G'' and η* at 0.1 % strain for the consumer study skin creams.*

CREAM	G′ at 0.1% strain		G″ at 0.1% strain		η* at 0.1% strain	
	Average	CV	Average	CV	Average	CV
	[Pa]	[%]	[Pa]	[%]	[Pa.s]	[%]
C1	1612	29.6	573	38.2	1711	30.5
C3	361	11.4	102	17.5	375	11.9
C5	9159	4.5	2978	8.1	9631	4.9
C8	635	55.2	162	60.6	655	55.6
C9	2995	12.6	1032	18.0	3168	13.1
C11R	41	10.9	7	17.2	42	11.0
C12R	14970	7.3	5031	9.5	15793	7.4
C16	2068	5.9	428	3.2	2111	5.8
C18	4053	11.6	921	11.6	4156	11.6
C27	11463	7.4	2905	8.3	11823	7.3
C28	958	9.9	397	8.0	1037	9.6
C32	17053	20.2	8757	24.0	19173	20.9

Table A11.3: *Oscillation amplitude sweep summary table including average and CV values for G', G" and η* at 1 % strain for the consumer study skin creams.*

CREAM	G′ at 1% strain		G″ at 1% strain		η* at 1% strain	
	Average	CV	Average	CV	Average	CV
	[Pa]	[%]	[Pa]	[%]	[Pa.s]	[%]
C1	1217	25.1	388	34.9	1277	25.9
C3	351	9.6	90	13.9	362	9.9
C5	8735	2.7	2512	5.2	9089	2.9
C8	573	52.6	127	61.6	587	53.0
C9	2601	11.4	801	15.1	2722	11.7
C11R	38	6.7	6	4.3	39	6.6
C12R	14964	4.8	4431	5.8	15607	4.9
C16	2015	4.0	359	3.3	2047	4.0
C18	4038	11.9	828	12.8	4123	11.9
C27	10834	5.4	2325	4.3	11080	5.4
C28	925	10.1	350	9.7	989	10.0
C32	9083	19.3	4028	23.2	9936	19.9

Table A11.4: *Oscillation amplitude sweep summary table including average and CV values for G', G" and η* at 100 % strain for the consumer study skin creams.*

CREAM	G′ at 100% strain		G″ at 100% strain		η* at 100% strain	
	Average	CV	Average	CV	Average	CV
	[Pa]	[%]	[Pa]	[%]	[Pa.s]	[%]
C1	110	16.8	95	20.8	145	18.5
C3	38	10.8	32	11.7	50	11.2
C5	1739	1.3	1288	2.4	2165	1.5
C8	57	37.2	59	41.9	83	39.6
C9	233	8.0	253	11.1	344	9.6
C11R	5	4.5	5	4.2	7	4.0
C12R	721	4.1	1037	3.1	1263	3.4
C16	609	4.9	375	3.6	716	4.4
C18	384	7.5	478	9.6	613	8.6
C27	1982	9.5	1401	6.4	2430	8.2
C28	108	9.1	114	7.3	157	8.1
C32	390	10.8	491	13.9	627	12.6

Table A11.5: *Oscillation amplitude sweep summary table including average and CV values for tanδ at 0.1 %, 1 % and 100 % strain for the consumer study creams.*

CREAM	tanδ at 0.1 % strain		tanδ at 1 % strain		tanδ at 100 % strain	
	Average [Pa]	CV [%]	Average [Pa]	CV [%]	Average [Pa.s]	CV [%]
C1	0.36	48.3	0.32	43.0	0.86	26.8
C3	0.28	21.0	0.26	16.9	0.83	16.0
C5	0.33	9.3	0.29	5.9	0.74	2.7
C8	0.26	82.0	0.22	81.0	1.04	56.0
C9	0.34	22.0	0.31	18.9	1.09	13.7
C11R	0.17	20.3	0.14	7.9	1.11	6.1
C12R	0.34	11.9	0.30	7.5	1.44	5.1
C16	0.21	6.8	0.18	5.2	0.62	6.1
C18	0.23	16.4	0.21	17.5	1.25	12.2
C27	0.25	11.1	0.21	6.9	0.71	11.4
C28	0.41	12.8	0.38	14.0	1.06	11.7
C32	0.51	31.3	0.44	30.2	1.26	17.6

Table A11.6*: Oscillation frequency sweep summary table including the magnitude of the slopes logG'-logω and logG"-logω, the intercepts and the R^2 values for the consumer study skin creams.*

CREAM	logG′-logω			logG″-logω		
	Slope	Intercept	R-squared	Slope	Intercept	R-squared
C1	0.111	3.186	0.992	0.083	2.680	0.963
C3	0.156	2.553	0.982	0.006	1.975	0.063
C5	0.158	3.946	0.997	0.118	3.419	0.998
C8	0.118	2.726	0.997	0.054	2.070	0.948
C9	0.162	3.496	0.998	0.141	2.993	0.999
C11R	0.101	1.593	0.956	0.306	0.786	0.940
C12R	0.177	4.165	0.989	0.038	3.645	0.854
C16	0.106	3.278	0.993	0.033	2.547	0.908
C18	0.133	3.610	0.999	0.081	2.934	0.958
C27	0.112	4.013	0.986	-0.018	3.351	0.987
C28	0.229	2.999	0.999	0.193	2.590	0.993
C32	0.134	3.971	1.000	0.099	3.614	0.987

Table A11.7: *Oscillation frequency sweep summary table including the average tanδ, G' and G" data at 1 rad.s^{-1} for the consumer study skin creams.*

CREAM	tanδ at 1 rad.s^{-1}		G′ at 1 rad.s^{-1}		G″ at 1 rad.s^{-1}	
	Average	CV	Average	CV	Average	CV
	[-]	[%]	[Pa]	[%]	[Pa]	[%]
C1	0.30	22.5	1561	16.0	471	15.9
C3	0.27	5.2	367	3.5	98	3.9
C5	0.29	6.6	8925	3.5	2615	5.6
C8	0.21	57.6	538	35.9	114	45.0
C9	0.31	18.7	3171	13.3	981	13.2
C11R	0.14	13.9	39	9.5	5	10.1
C12R	0.30	5.8	15033	4.8	4480	3.3
C16	0.18	4.3	1917	2.5	351	3.6
C18	0.21	18.7	4097	12.0	842	14.3
C27	0.21	6.1	10500	4.2	2237	4.5
C28	0.38	9.6	1010	6.2	383	7.4
C32	0.43	42.5	9383	25.2	4023	34.2

Table A11.8: *Cross model summary table containing the infinite and zero shear viscosities for the consumer study skin creams.*

	Infinite Shear Viscosity			Zero Shear Viscosity		
CREAM	Average	Median	CV	Average	Median	CV
	[Pa.s]	[Pa.s]	[%]	[Pa.s]	[Pa.s]	[%]
C1	0.131	0.123	29.5	31145	28626	14.1
C3	0.269	0.258	7.6	10542	10443	7.6
C5	0.055	0.061	88.2	216860	214960	3.8
C8	0.177	0.186	13.8	15414	17034	28.4
C9	0.090	0.086	10.7	89120	90502	9.3
C11R	0.265	0.257	7.3	8186	8587	24.5
C12R	0.316	0.224	50.4	322697	334460	6.3
C16	0.241	0.277	27.0	87443	89466	15.9
C18	0.109	0.109	4.6	155550	157550	15.4
C27	0.089	0.000	173.2	299050	301290	1.4
C28	0.119	0.119	1.6	17581	16811	8.5
C32	0.034	0.049	86.7	241797	225350	36.8

Table A11.9: *Cross model summary table containing the cross model a and p-values for the consumer study skin creams.*

	CROSS model a value			CROSS model p value		
SAMPLE	**Average**	**Median**	**CV**	**Average**	**Median**	**CV**
	[s]	**[s]**	**[%]**	**[-]**	**[-]**	**[%]**
C1	200	192	12.5	0.906	0.912	1.59
C3	171	168	4.8	0.867	0.862	1.13
C5	90	89	1.7	0.934	0.934	0.05
C8	211	206	15.3	0.897	0.898	2.85
C9	228	233	5.5	0.915	0.917	0.42
C11R	815	854	28.8	0.715	0.717	0.78
C12R	195	195	0.3	0.993	0.987	1.02
C16	103	104	4.3	0.959	0.959	0.23
C18	279	270	6.6	0.950	0.951	0.31
C27	118	120	4.7	0.978	0.978	0.14
C28	115	117	4.6	0.938	0.938	0.80
C32	393	378	18.6	0.908	0.907	1.12

Table A11.10: *Steady shear summary table containing the cross model R-squared values and the yield stresses for the 12 consumer study creams. Note that the shear rate ranged between 0.0001 and 10,000 s^{-1} for all samples.*

	CROSS MODEL R-SQUARED VALUES	YIELD STRESS		
SAMPLE	**Average**	**Average**	**Median**	**CV**
	[-]	**[Pa]**	**[Pa]**	**[%]**
C1	0.996	97	96	2.8
C3	0.994	30	31	7.2
C5	0.997	1092	1133	8.7
C8	0.996	37	34	31.0
C9	0.997	206	206	2.8
C11R	0.988	4	4	3.0
C12R	0.990	783	798	8.3
C16	0.999	447	454	15.6
C18	0.994	263	280	16.0
C27	0.999	1248	1239	2.6
C28	0.997	86	87	8.2
C32	0.979	295	287	29.7

Appendix XII: Predictive models including rheology and force plate analysis parameters

Table A12.1: *Predictive model equations and goodness of fit data for predicting sensory scores from force plate analysis and rheological parameters.*

Attribute	Final model equations	R^2	Adjusted R^2	Predicted R^2
PC2				
DRYING	Log(Drying) = +3.193 -0.804 * logG″ at 100 % strain +5.739 * log(Coeff) factor E1	0.893	0.869	0.825
DRAGGING	Log(Dragging) = +2.488 -0.572 * lgG" at 100% strain +4.397 * lg(Coeff) factor E1	0.859	0.828	0.769
FINAL GREASINESS	Final Greasiness = -11.695 +4.372 * logG″ at 100 % strain -32.905 * log(Coeff) factor E1	0.845	0.811	0.721
ABSORPTION	Absorption = -5.458 +3.783 * logG″ at 100 % strain -20.225 * log(Coeff) factor E1	0.801	0.756	0.664